WAL TR 766.2/3-4

FIFTH QUARTERLY PROGRESS REPORT
EVALUATION OF HIGH-STRENGTH LIGHTWEIGHT LAMINATED PRESSURE VESSELS OF LAP-JOINT CONSTRUCTION

by

G. Citrin

(RAC 1313, 244-3005)

10 April 1963

Period covered: 1 January to 31 March 1963
Republic Aviation Corporation
Farmingdale, L. I., N. Y.

Contract DA-30-069-ORD-3440

New York Procurement District, U. S. Army

00 Project No. -OMS Code 5010.11.8430051

Department of the Army Project No. 59332008
Watertown Arsenal
Watertown 72, Massachusetts

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The findings in this report are not to be construed as an official Department of the Army position.

Rockets, Motor cases
Adhesives
Joining

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ABSTRACT

FIFTH QUARTERLY PROGRESS REPORT EVALUATION OF HIGH-STRENGTH, LIGHTWEIGHT LAMINATED PRESSURE VESSELS OF LAP-JOINT CONSTRUCTION

During this quarter, five pressure vessels were assembled and tested to failure. They were fabricated of three nominal thicknesses of material, 0.025-, 0.040-, and 0.064-inch-thick mar-aging steel. An analysis of the results of these tests indicated the feasibility of the lightweight laminated pressure vessels of lap-joint design and showed the difficulty of demonstrating a reproducible confidence level with reusable header closures that had sustained some deformation during hydrostatic tests to high-energy levels.

SECTION I

INTRODUCTION

A. PURPOSE

This contract is being performed to demonstrate the basic feasibility and the achievable confidence level of the overlapping cylinder design as applied to the production of high-strength pressure vessels made of high-nickel, mar-aging steels. Three nominal thicknesses of material, 0.025, 0.040, and 0.064 inch, are being used for the evaluation of the overlapping cylinder design on pressure vessels having wall thicknesses of 0.050, 0.080, and 0.128 inch, respectively. The pressure vessels will be nominally 24 inches in diameter and from 40 to 90 inches long. Phases 1 and 2 are investigations of structural adhesives and of brazed or ceramic bonded joints, respectively. Republic is working only on Phase 1. Phase 2 has been deleted from the contract during this quarter.

B. SUMMARY OF ACTIVITIES COVERED BY PREVIOUS REPORTS

The effort during the first quarter, which was primarily on the materials survey, resulted in a tentative selection of the 18-percent-nickel, mar-aging steels as having the greatest potential usefulness. Small quantities of sample materials were evaluated. The basic weldability characteristics were found to be excellent, and the potential of these alloys for parent-metal operation at the 300,000-psi yield-strength levels was confirmed. Preliminary tests also indicated satisfactory bonding characteristics. The results of these probing tests were sufficiently encouraging to warrant ordering adequate test quantities of both the 18-7-5 (INCO 250 KSI) and 18-9-5 (INCO 300 KSI) NiCoMo, production-type alloys.

During the second quarter, the test quantities of material were received and evaluated sufficiently to ensure their suitability for this program. A final selection of the 18-9-5 (INCO 300 KSI) composition was made, and the pressure-vessel material was ordered.

During these two periods, the tool design and a major portion of the tool fabrication were completed. The lap-shear test program for adhesive selection

was started, and preliminary subscale ring-sizing tests were successfully performed using welded preforms made of the 18-9-5 NiCoMo alloy.

During the third quarter, the lap-shear testing program for the selection of adhesives was completed. The designs for the work-horse headers were also completed. Header-machining and heat-treat operations were started.

The remaining subscale ring-sizing tests were performed, and the necessary allowances for variables, such as spring back and subsequent aging shrinkage, were established to ensure dimensional control.

The effort during the fourth quarter was expended on resolving the mar-aging steel production problems at the mill and on fabricating and testing the first pressure vessel.

The first partial delivery of production material (received in September 1962) did not comply with several of the specification requirements. Therefore, it was sent back to the mill for reprocessing. Usable production materials with reduced thicknesses were finally available in the middle of November 1962.

In December, assembly of the first pressure vessel was started using 0.021-inch-thick steel (originally 0.025-inch thick) and polyamide-epoxy (FM1000) adhesive. This vessel was hydrostatically tested; it burst at 990 psig internal pressure and withstood a hoop stress of 286,000 psi in the mar-aging steel wall. There was no failure or permanent deformation in the adhesive joint, and the cylinder weldments showed no primary failures.

C. SUMMARY OF ACTIVITIES COVERED BY THIS REPORT

During the fifth quarter, five assemblies were fabricated and tested to failure. A detailed analysis of the test results is presented in this report. A short summary of the results of tests conducted in the program is shown in Table 1. Vessel III-101 was included in the tabulation for information only; this vessel was assembled and tested in the fourth quarter.

Of the five vessels fabricated and tested during this quarter, three vessels had metal failures and two had adhesive failures. The adhesive failures have both been attributed to the use of a work-horse reusable header design in which the skirt section is required to be substantially thicker than the mar-aging steel

TABLE 1. SUMMARY OF EVALUATION TEST RESULTS

Vessel Assembly Number	Test Number	Total Metal Wall Thickness (inch)	Adhesive	Burst Pressure (psi)	Stress at Failure (psi)	Remarks
III-101*	1	0.041	FM1000	990	286,000	Failed in parent metal at a stress level above uniaxial strength of the mar-aging steel. Failure not associated with weld or adhesive joint.
III-102**	2	0.041	FM47	250	--	Low-pressure leak caused by inability of this adhesive to compensate for a poor fit-up condition caused by set in the work-horse headers. This adhesive was dropped from further investigation.
V-103**	3	0.041	FM1000	500	144,000	Failure initiated in the parent metal adjacent to a header. Metallurgical examination disclosed a local flaw at the origin of the failure.
V-201**	4	0.075	FM1000	2030	320,000	Failure originated in parent metal and was not associated with the weldment or adhesive. The stress level exceeded by 12 percent the uniaxial tensile strength of the mar-aging steel used.
V-202**	5	0.075	FM1000	1520	240,000	Failure originated in a weldment in the central inner ring. Examination showed no unusual conditions in the weld. Uniaxial tensile strengths of weldments tested range from 243,000 to 263,000 psi.
V-301**	6	0.124	FM1000	2290	219,000	Failure due to unbonded area in one header joint (approximately 20 percent of area). Preliminary evaluation attributed this condition to the stiffness of the work-horse header preventing uniform contact pressure during the cure cycle.

* The detailed report on vessel III-101 is in the "Fourth Quarterly Progress Report," RAC 1160.
** See Section III.

rings making up the cylindrical portion of the vessel. The resultant stiffness prevents uniform cure pressure in the faying surfaces. In addition, the headers do sustain a small amount of permanent set due to the design yield strength corresponding to the 0.2-percent offset value rather than to the proportional limit.

D. SUMMARY OF ACTIVITIES PLANNED FOR NEXT QUARTER

During the next quarter, fabrication and test of additional pressure vessel assemblies will continue; emphasis will be placed on overcoming the drawbacks of the work-horse header design in order to obtain reproducible assemblies for test purposes.

SECTION II

MATERIAL USED

A. GENERAL

During the fifth quarter, the major effort has been the manufacture, test, and evaluation of pressure-vessel assemblies. As part of this work, the development of revised welding schedules using supplementary filler wire has been completed. This was necessary due to the results obtained in ring-sizing tests with the production material. A limited number of tensile specimens was tested to aid in the evaluation of the pressure-vessel tests, and a detailed metallurgical investigation was conducted on vessel V-103 because of a low-stress parent-metal failure. The results of the welding and metallurgical work are discussed under the appropriate subject headings.

B. WELDING/METALLURGICAL EVALUATION

As previously reported, the technique for using run-off tabs at the start and end of the welds in order to eliminate the necessity of a trim operation was developed (see Figure 1). The technique that proved most satisfactory on test work pieces was starting the weld at a point on the ring approximately 0.060 inch from the edge. By the proper adjustment of travel-start delay and the addition of a small amount of filler wire, the weld puddle is forced to flow back to the edge of the ring and is fused to the run-off tab. When attempting cylinder manufacture, it became apparent that, because of the high degree of precision fit-up required and the inherent spring-like character of the cold-rolled cylinders, the positioning operation would be too tedious and time consuming to produce sets of welded rings reliably and economically. In fact, the time spent in positioning the cylinder and end tabs nullified the savings gained by eliminating the trim operation. Accordingly, a 0.5-inch trim allowance was provided to simplify the positioning of the cylindrical rings and the starting and stopping of the weld sequence. The welding schedule for the 0.025-inch (nominal) thick mar-aging steel is shown in Table 2.

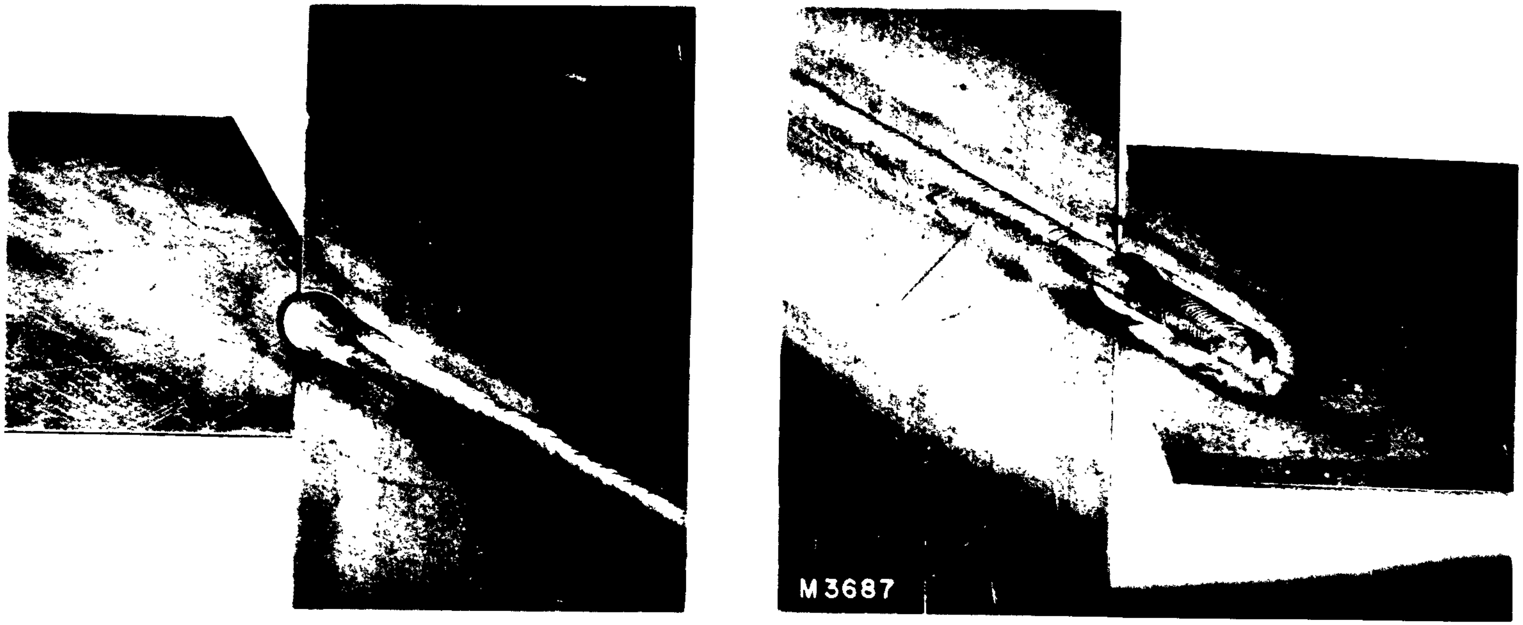


Figure 1. Welding Run-off Tab Configuration

TABLE 2. T.I.G. WELDING SCHEDULE FOR 0.025 (NOMINAL) -INCH-THICK INCO 300-KSI COLD-ROLLED SHEET ALLOY

Item	Condition
Sciaky model FH8-96 (power source)	
Current	30 amperes dc, straight polarity
Voltage	6.0 to 7.0 volts, high-frequency arc initiation
Water-cooled torch (Airco M50B)	
Electrode	0.040-inch diameter, 2-percent thoriated, blunt taper
Arc gap	0.040 inch
Nozzle	Copper, 0.375-inch inside diameter
Shielding	Argon gas, 6 cfh
Tungsten extension	0.250 inch below bottom of nozzle
Torch angle	90 degrees to work
Travel speed	5 ipm
Filler metal	None
Fixture	
Backup bar	Copper, no coolant
Hold-down bar	Copper, line-clamping contact
Groove shape	Rectangular
Groove width	0.125 inch
Groove depth	0.040 inch
Hold-down pressure	40 psi, pneumatic pressure
Hold-down spacing	0.187 inch
Joint gap	0.002 inch, maximum
Backup shielding	Argon gas, 6 cfh

As the ring-sizing operation progressed, it was determined that weld reinforcement was required in order to be able to size the cylinders sufficiently to maintain the desired permanent set required to obtain full roundness. Therefore, the weld schedules previously developed were modified slightly (see Tables 3 and 4) to provide for the addition of filler metal.

The 0.020-inch and 0.040-inch-diameter wire originally purchased proved to be too large to develop a suitable schedule to meet the maximum bead height (0.004 inch high) specified for compatibility with the adhesive bonding technique. In anticipation of this possibility, approximately 5 pounds of the 0.040-inch-diameter wire had been redrawn to a 0.010-inch diameter. This lighter gage wire made it possible to develop suitable schedules for the 0.040- and 0.064-inch (nominal) thich mar-aging steel rings (see Tables 4 and 5).

A number of the 0.040-inch nominal gage cylinders was welded without filler prior to the establishment of the need for weld reinforcement (see Table 3). These cylinders were subsequently rewelded with a second pass using filler wire and the schedule shown in Table 4.

A mechanical-property investigation was conducted on a limited number of specimens taken from production mar-aging sheet material to confirm the designated aging heat treatment. The data are presented in Table 6. The group of specimens designated by superscripts c, d, and e accompanied the respective rings (used in the assemblies noted in Table 6) during heat treatment in a production electric muffle furnace (certified by the quality control laboratory to ±10 degrees F). The specimens designated by the superscript b were aged in a small laboratory electric muffle furnace with thermocouples attached to the specimens. The aging response of the laboratory control specimens was 5 percent higher than the production control specimens. The strength values achieved with both the production and laboratory controls are well within the requirements for this heat of material.

A metallurgical investigation was conducted in order to determine the cause of failure of pressure vessel V-103. The origin of failure was determined visually, and a section was taken from this area for metallographic analysis. The study involved the examination and removal of successive layers of material to approach the origin in small enough increments to preserve and reveal any unusual structure.

TABLE 3. T.I.G. WELDING SCHEDULE FOR 0.040 (NOMINAL) -INCH-THICK INCO 300-KSI COLD-ROLLED SHEET ALLOY

Item	Condition
Sciaky model FH8-96 (power source)	
Current	45 amperes dc, straight polarity
Voltage	6.5 volts, high-frequency arc initiation
Water-cooled torch (Airco M50B)	
Electrode	0.040-inch diameter, 2-percent thoriated, blunt taper
Arc gap	0.040 inch
Nozzle	Copper, 0.375-inch inside diameter
Shielding	Argon gas, 6 cfh
Tungsten extension	0.250 inch below bottom of nozzle
Torch angle	90 degrees to work
Travel speed	5 ipm
Filler metal	None
Fixture	
Backup bar	Copper, no coolant
Hold-down bar	Copper, line-clamping contact
Groove shape	Rectangular
Groove width	0.250 inch
Groove depth	0.040 inch
Hold-down pressure	40 psi, pneumatic pressure
Hold-down spacing	0.281 inch
Joint gap	0.002 inch, maximum
Backup shielding	Argon gas, 8 cfh

TABLE 4. T. I. G. WELDING SCHEDULE FOR 0.040 (NOMINAL) -INCH-THICK INCO 300-KSI COLD-ROLLED SHEET ALLOY WITH FILLER WIRE

Item	Condition
Sciaky model FH8-96 (power source)	
Current	48 amperes dc, straight polarity
Voltage	6.5 volts, high-frequency arc initiation
Water-cooled torch (Airco M50B)	
Electrode	0.040-inch diameter, 2-percent thoriated, blunt taper
Arc gap	0.040 inch
Nozzle	Copper, 0.375-inch inside diameter
Shielding	Argon, 6 cfh
Tungsten extension	0.250 inch below bottom of nozzle
Torch angle	90 degrees to work
Travel speed	5 ipm
Filler metal	18-percent nickel mar-aging steel, 0.010-inch diameter
Wire speed	32 ipm
Fixture	
Backup bar	Copper, no coolant
Hold-down bar	Copper, line-clamping contact
Groove shape	Rectangular
Groove width	0.250 inch
Groove depth	0.040 inch
Hold-down pressure	40 psi, pneumatic pressure
Hold-down spacing	0.281 inch
Joint gap	0.002 inch, maximum
Backup shielding	Argon gas, 8 cfh

TABLE 5. T. I. G. WELDING SCHEDULE FOR 0.064 (NOMINAL) -INCH-THICK INCO 300-KSI COLD-ROLLED SHEET ALLOY WITH FILLER WIRE

Item	Condition
Sciaky model FH8-96 (power source)	
Current	80 amperes dc, straight polarity
Voltage	6.5 volts, high-frequency arc initiation
Water-cooled torch (Airco M50B)	
Electrode	0.60-inch diameter, 2-percent thoriated, blunt taper
Arc gap	0.040 inch
Nozzle	Copper, 0.375-inch inside diameter
Shielding	Argon, 10 cfh
Tungsten extension	0.250 inch below bottom of nozzle
Torch angle	90 degrees to work
Travel speed	5 ipm
Filler metal	18-percent nickel mar-aging steel, 0.010-inch diameter
Wire speed	32 ipm
Fixture	
Backup bar	Copper, no coolant
Hold-down bar	Copper, line-clamping contact
Groove shape	Rectangular
Groove width	0.250 inch
Groove depth	0.040 inch
Hold-down pressure	40 psi, pneumatic pressure
Hold-down spacing	0.281 inch
Joint gap	0.002 inch, maximum
Backup shielding	Argon, 12 cfh

TABLE 6. MECHANICAL PROPERTIES OF PRODUCTION MATERIAL

Specimen Condition	18-9-5 NiCoMo (300-KSI) Mar-Aging Steel Specimen Characteristics					
	Load Direction[a]	Nominal Thickness (inch)	0.2-Percent Offset Yield Strength (KSI)	Ultimate Tensile Strength (KSI)	Elongation (percent)	
					in 1 inch	in 2 inches
As received and aged at 925 ±10 degrees F for 3 hours[b]	T	0.025	278.2	287.7	2.0	3.0
	T	0.025	276.4	286.4	1.5	2.0
	T	0.025	275.2	284.4	1.5	3.0
	T	0.025	272.2	280.8	2.0	3.0
	T	0.025	273.9	283.9	1.5	2.0
	T	0.025	273.9	281.9	1.5	2.0
	T	0.040	289.3	299.5	2.5	4.0
	T	0.040	292.0	297.9	2.5	4.0
	T	0.040	294.1	302.2	2.0	3.0
As received and aged at 925 ±10 degrees F for 3 hours in production electric muffle furnace (quality-control certified to ±10 degrees F)	L[c]	0.040	285.8	290.2*	1.5	3.0
	L[c]	0.040	269.1	282.9*	1.5	3.0
	L[c]	0.040	266.7	283.3*	1.5	3.0
	L[d]	0.040	277.4	281.9*	1.5	3.0
	L[d]	0.040	253.9	285.3*	1.5	3.0
	L[d]	0.040	255.0	285.3*	1.5	3.0
	L[d]	0.040	279.1	283.2*	1.5	3.0
	L[e]	0.064	304.1	309.2	2.5	5.0
	L[e]	0.064	304.4	307.6	2.5	5.0
	L[e]	0.064	305.4	308.5	1.5	3.0
As received and aged at 900 ±10 degrees F for 3 hours[b]	L	0.064	307.5	311.4	2.0	4.0
	L	0.064	305.4	308.7	2.0	4.0
	L	0.064	303.7	307.7	2.5	5.0
As received and aged at 925 ±10 degrees F for 3 hours[b]	L	0.064	320.1	324.0	2.5	5.0
	L	0.064	323.6	326.9	2.5	5.0
	L	0.064	321.4	324.7	2.5	5.0

(a) T is transverse and L is longitudinal relative to the grain of the metal
(b) Control specimens heat treated in small laboratory electric muffle furnace with thermocouple on specimens
(c) Control specimens accompanying rings used in assembly V-201 during heat treatment
(d) Control specimens accompanying rings used in assembly V-203 during heat treatment
(e) Control specimens accompanying rings used in assembly V-301 during heat treatment
* Average ultimate test strength of starred items is 284.6

From the data obtained, the cause of failure can be attributed to the following factors:

1) The presence of multiple discontinuities in the form of grooves and notches on the surface of the material

2) The presence of an isolated zone of light-etching, hard material of unknown origin

In addition to these defects, metallographic examination and microhardness determinations revealed a soft, white outer layer (0.0001 to 0.0003 inch thick) that surrounded a major portion of the outer periphery of the original surface. This layer, which followed the irregular grooved contour of the surface, was attributed to oxidation and/or depletion due to improper mill processing. The layer was readily removed by sand blasting; it could easily be reintroduced on exposure to the solution annealing temperature.

Figure 2 shows the location of the fracture origin relative to the secondary fracture as well as the relative location of the photomicrographs. Figure 3 shows the light-etching, hard zone devoid of structure. Evidence of the softer layer previously discussed is presented in Figures 4 and 5. Figure 6 shows the discontinuous nature of this layer.

The appearance of the microstructure in Figure 3 is noteworthy. A microhardness traverse indicated that the lightly etched area contained two zones. The first was an outer soft layer from 0.0001 to 0.0003 inch in depth ranging in hardness from R_b (Rockwell b scale) 84 to R_c (Rockwell c scale) 29. The second area was harder (R_c 60 to 61) and extended to a depth of 0.006 inch. This corresponded to the inner extremity of the fracture zone; the darker etched field originated at this point. Hardness values were found to be R_c 57; these remained constant to the midpoint of the section. Beyond this point, the values decreased to R_c 50 and remained at this level to the opposite surface.

Before polishing to the level of the origin, the microstructure was uniformly attacked by the etchant; microhardness traverses indicated completely uniform hardness values (R_c 54) from surface to surface. Below the origin, the fracture zone became increasingly less evident (see Figure 7); no fracture can be seen in Figure 8. A microhardness traverse across the area shown in Figures 7 and 8 indicated no appreciable difference in hardness (R_c 54).

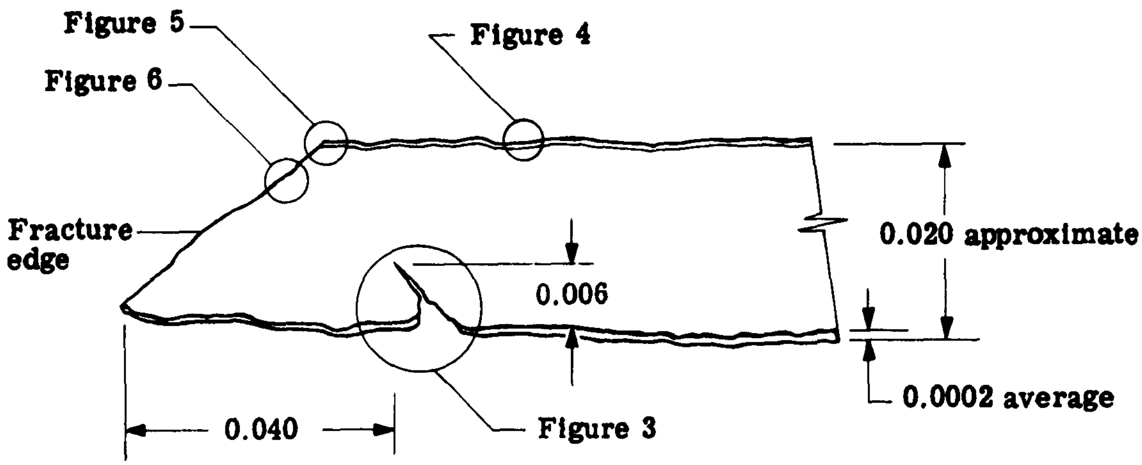


Figure 2. Edge View of Failure Origin and Locations of the Micrographs

Figure 3. Secondary Fracture in 18-9-5 Mar-aging Steel (about 250X, Schantz Etch)

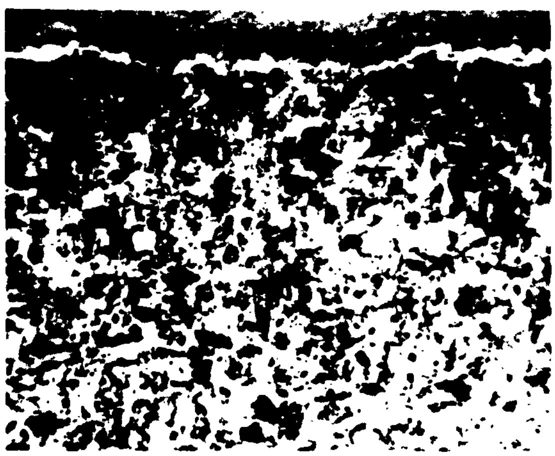


Figure 4. 18-9-5 Mar-aging Steel (about 750X, Schantz Etch)

Figure 5. 18-9-5 Mar-aging Steel Adjacent to Fracture (about 1000X, Schantz Etch)

Figure 6. Fracture Edge of 18-9-5 Mar-aging Steel (about 1000X, Schantz Etch)

Figure 7. Secondary Fracture of 18-9-5 Mar-aging Steel (about 250X, Schantz Etch)

Figure 8. Next Section below Area Shown in Figure 7. No Secondary Fracture Evident (about 750X, Schantz Etch)

To duplicate the softer white layer in order to understand its possible cause, three additional sections from the assembly were sand blasted to remove all traces of the previously found white layer. These specimen sections were then solution annealed (1500 degrees F for 30 minutes) and aged (925 degrees F for 3 hours) under controlled conditions. The first specimen was solution annealed in a vacuum furnace and argon quenched to room temperature. The second specimen was solution annealed in an electric muffle furnace and quenched in liquid nitrogen. The third specimen was solution annealed in the same manner and air quenched. All three specimens were then aged in the same furnace.

Figures 9, 10, and 11 show the results of these processes. In each case, a corner of the specimen is shown, the microstructure of which is typical of that over the entire section. Clearly, atmosphere was an important factor in the formation of this layer. In both cases where no protective atmosphere was provided, an efficient, tightly adherent outer layer occurred that was extremely soft. Hardness readings in this area varied from R_b 72 to R_c 33.

The white layer is attributed to a combination of scale and/or a high nickel-iron depleted in cobalt or molybdenum. The International Nickel Company has reported observing a similar effect. Further liaison is being maintained to establish the nature of this layer.

C. FORMABILITY EVALUATION

No further evaluation work was performed with respect to the formability of the mar-aging steel. The information developed during the previous reporting periods is sufficient to permit the manufacture of rings in all three gages with the proper dimensional control and stability. Rings were manufactured for the V-201, V-202, V-203, and V-301 assemblies during this quarter. The dimensions of the rings are tailored for each particular assembly due to the deformation sustained by the headers after each use. The results of this ring fabrication are shown in Table 7.

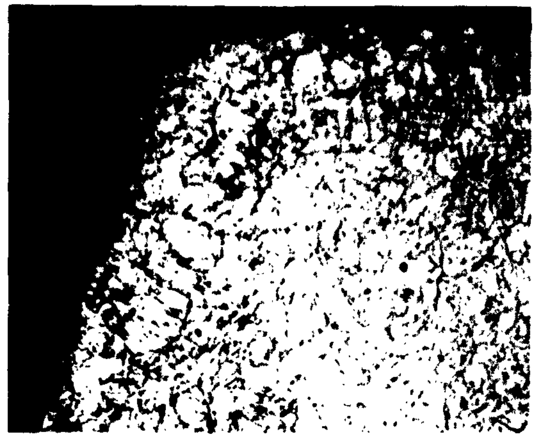


Figure 9. 18-9-5 Mar-aging Steel, Re-solution Treated in Vacuum Furnace and Argon Quenched (about 750X, Schantz Etch)

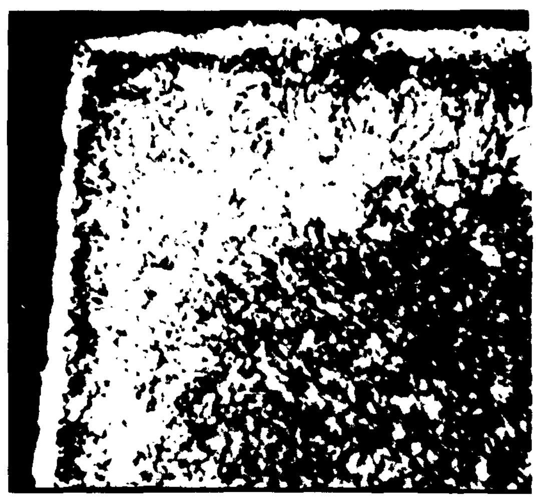


Figure 10. 18-9-5 Mar-aging Steel, Re-solution Treated in Electric Muffle Furnace and Liquid-Nitrogen Quenched (about 750X, Schantz Etch)

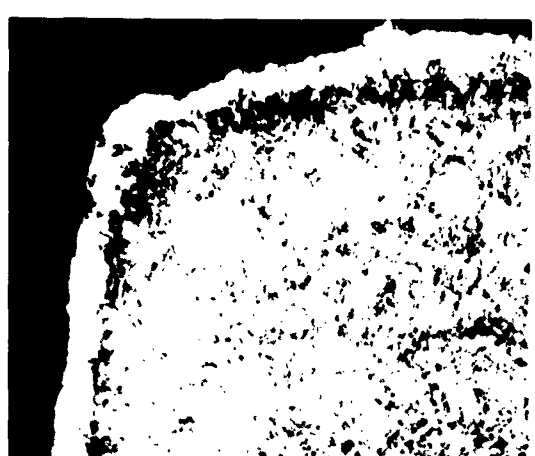


Figure 11. 18-9-5 Mar-aging Steel, Re-solution Treated in Electric Muffle Furnace and Air Quenched (about 750X, Schantz Etch)

TABLE 7. SUMMARY OF RESULTS OF RING FABRICATION

Assembly Number	Part Number*	Preform Diameter** (inches)	Total Deformation (percent)	Total Permanent Set (percent)	Diameter before Aging** (inch)	Shrinkage in Aging (inch)	Diameter after Aging** (inches)
V-201	4-22-6-A	23.492 O	1.75	1.0	23.717 O	0.018	23.699 O
	4-22-1-A	23.475 O	1.75	1.0	23.717 O	0.019	23.698 O
	4-22-9-A	23.489 O	1.75	1.0	23.719 O	0.019	23.700 O
	4-23-1-B	23.453 I	2.0	1.12	23.726 I	0.021	23.705 I
	4-23-6-B	23.442 I	2.0	1.12	23.726 I	0.020	23.706 I
V-202	4-12-2-B	23.523 I	1.25	0.75	23.713 I	0.018	23.695 I
	4-11-5-B	23.537 I	1.25	0.75	23.715 I	0.019	23.696 I
	4-11-3-A	23.520 O	1.25	0.75	23.699 O	0.020	23.679 O
	4-11-4-A	23.520 O	1.25	0.75	23.696 O	0.019	23.677 O
	4-11-2-A	23.520 O	1.25	0.75	23.695 O	0.019	23.676 O
V-203	4-23-10-B	23.510 I	1.75	1.0	23.732 I	0.020	23.712 I
	4-12-5-B	23.513 I	1.75	1.0	23.730 I	0.016	23.714 I
	4-17-1-A	23.500 O	1.8	0.80	23.710 O	0.018	23.692 O
	4-12-4-A	23.452 O	1.8	1.0	23.711 O	0.019	23.692 O
	4-17-2-A	23.500 O	1.8	1.0	23.721 O	0.022	23.699 O
V-301	6-1-3-B	23.522 I	1.6	0.80	23.719 I	0.021	23.698 I
	6-1-2-B	23.510 I	1.6	0.80	23.720 I	0.020	23.700 I
	6-1-5-A	23.520 O	1.0	0.75	23.699 O	0.020	23.679 O
	6-2-2-A	23.521 O	1.0	0.75	23.700 O	0.020	23.680 O
	6-2-1-A	23.524 O	1.0	0.75	23.696 O	0.020	23.676 O

* A = inner ring
B = outer ring

** I = inside diameter
O = outside diameter

SECTION III

DESIGN AND TEST

A. INTRODUCTION

During the fifth quarter, five pressure vessel assemblies were tested. The vessels were fabricated in three gages of sheet metal material (0.021-, 0.038-, and 0.062-inch thick mar-aging steel rings). The configurations are shown in Figure 12, and the dimensions are tabulated in Table 8.

A permanent record of pressurization for each test was made. Figure 13 represents a typical pressure trace (test of vessel V-201 at a burst pressure of 2030 psi) as recorded on an oscillograph. The vertical timing traces are spaced at 0.1-second intervals. The hydraulic test console and pressurization chamber are shown in Figures 14 and 15. The test console has been modified for tests at pressures to 3800 psi, which is sufficient to test the vessels manufactured under this contract. Dial gages in the upper left the of console are used to monitor the pressure during the test. The test fixture visible inside the chamber includes swinging steel plates that function as blast deflectors during the high-energy burst tests. (One plate has been removed so that the vessel can be seen in its test position.) The hydraulic cylinder at the top of the fixture is used to catch the header after failure. The piston is attached to the header, and the upper portion of the cylinder is vented to the atmosphere so that the upward motion of the header is not restricted.

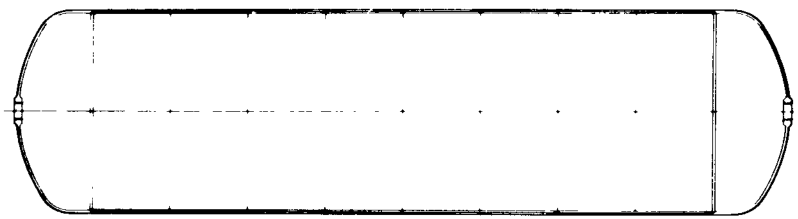
The discussion and evaluation of each of the tests follows in chronological order.

B. VESSEL 2

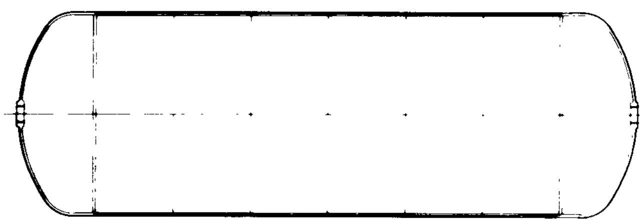
Vessel 2 (III-102) was fabricated using headers 244-1005-1 and 244-1006-1 (see Figures 16 and 17), FM47 adhesive, and three 0.021-inch-thick rings. These headers had been used in the III-101 assembly, and they had sustained permanent deformation. Figure 18 shows the dimensions of the headers before and after the test of assembly III-101. Header 244-1005-1 had been used in pressure vessel tests before this contract. The yield strength of this header was lower than the

Notes:

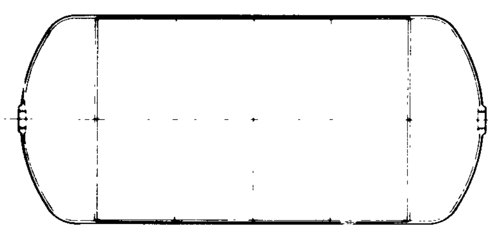
1. Butt gap, 0.040 inch (maximum)
2. A (outside diameter for inner ring) and B (inside diameter for outer ring) are sizing dimensions for detail parts
3. During assembly, rings are to be oriented to a minimum 4-inch peripheral spacing between welds
4. Surface preparation for adhesive bonding:
   0.064-inch rings. sand blast. 40 grit
   0.040 and 0.025-inch rings, liquid hone, 240 grit
   Maximum material removed from surface, 0.0005 inch
5. Weld line butt surface finish prior to welding, 32 rms



Configuration VII
Consists of 2 headers, 2 head inner rings, 2 body inner rings, and 3 outer rings



Configuration V
Consists of 2 headers, 2 head inner rings, 1 body inner ring, and 2 outer rings



Configuration III
Consists of 2 headers, 2 head inner rings, and 1 outer ring

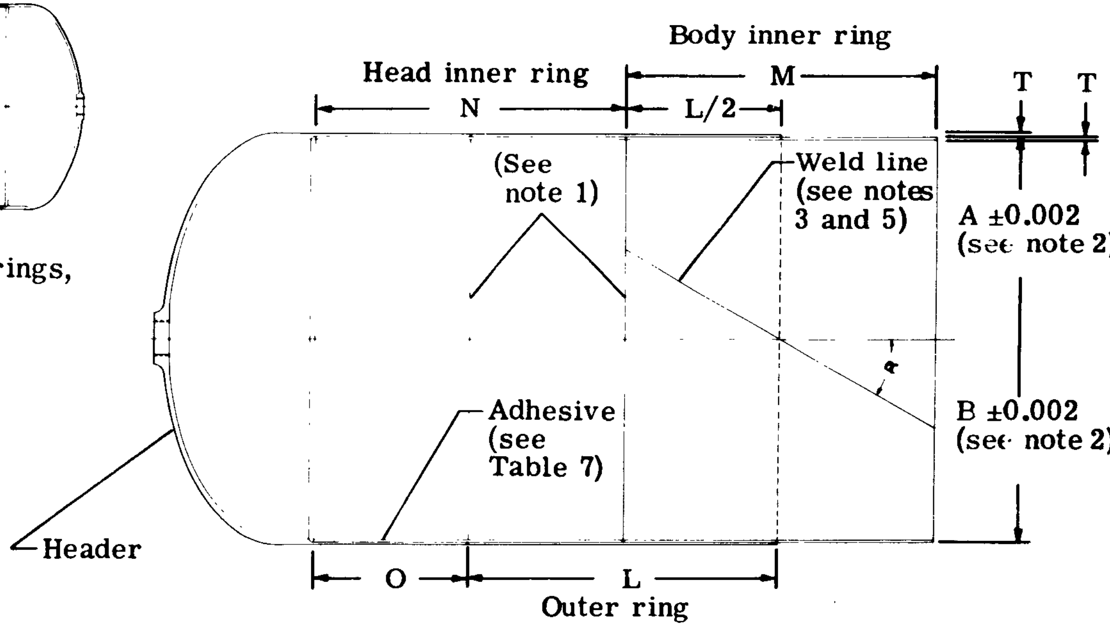


Figure 12. Test-Pressure-Vessel Configuration

TABLE 8. TEST-PRESSURE-VESSEL DATA

Assembly Dash Number	Configuration	Adhesive	Header Dash Number	Dimension (inches)*							Weld Line Angle, α (degrees)
				A	B	L	M	N	O	T	
101	III	FM1000	-1	23.715	23.715	10.00	--	11.00	6.00	0.021	30
102	III	FM47	-1	23.670	23.700	10.00	--	11.00	6.00	0.021	30
103	V	FM1000	-3	23.700	23.700	10.00	10.00	11.00	6.00	0.021	30
201	V	FM1000	-3	23.700	23.700	12.00	12.00	12.96	6.96	0.038	30
202	V	FM1000	-3	23.680**	23.695**	12.00	12.00	12.96	6.96	0.038	30
203***	V	FM1000	-3	23.692	23.712	12.00	12.00	12.96	6.93	0.038	30
301	V	FM1000	-5	23.680	23.700	16.00	16.00	16.96	8.96	0.062	30

* See Figure 12 for a description of the dimensions

** Dimension modified for use with -5 headers; the -3 headers were reused with extra adhesive in the header-to-ring joint because the -5 headers were not available

*** Rings manufactured, but vessel not assembled during this quarter

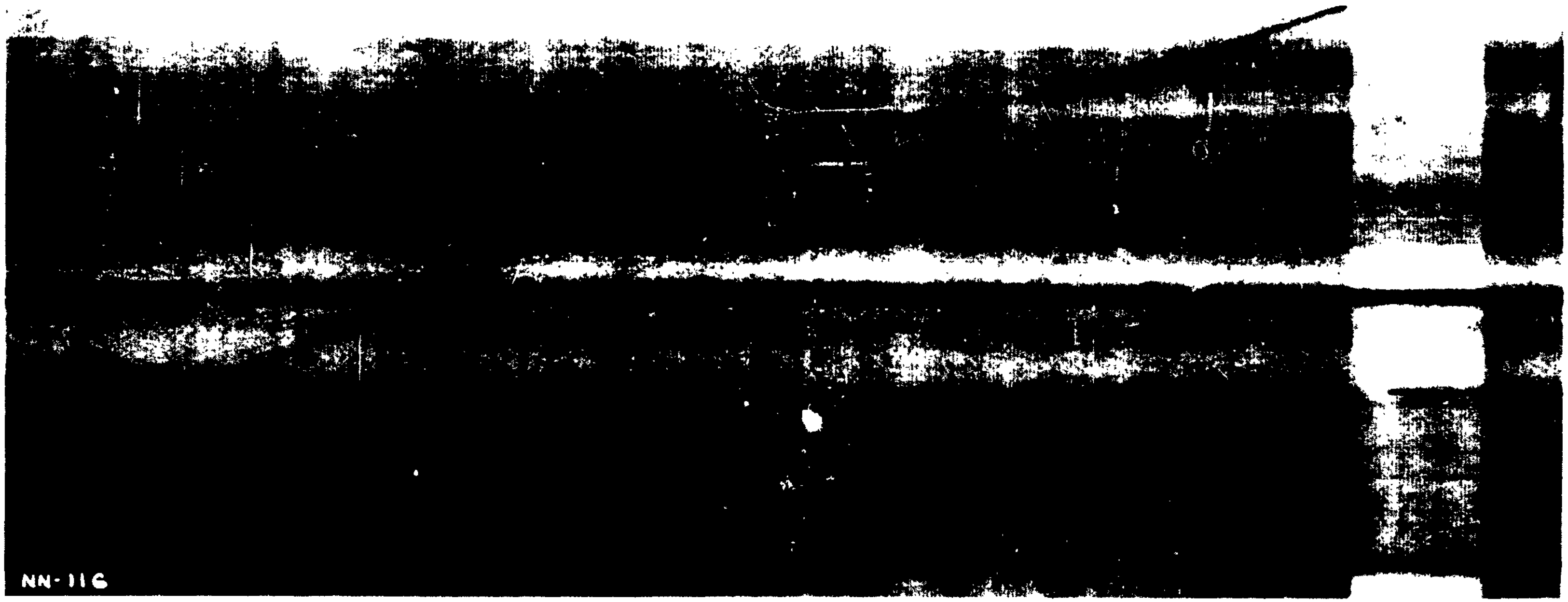


Figure 13. Pressure Trace Recorded during Test of Vessel V-201

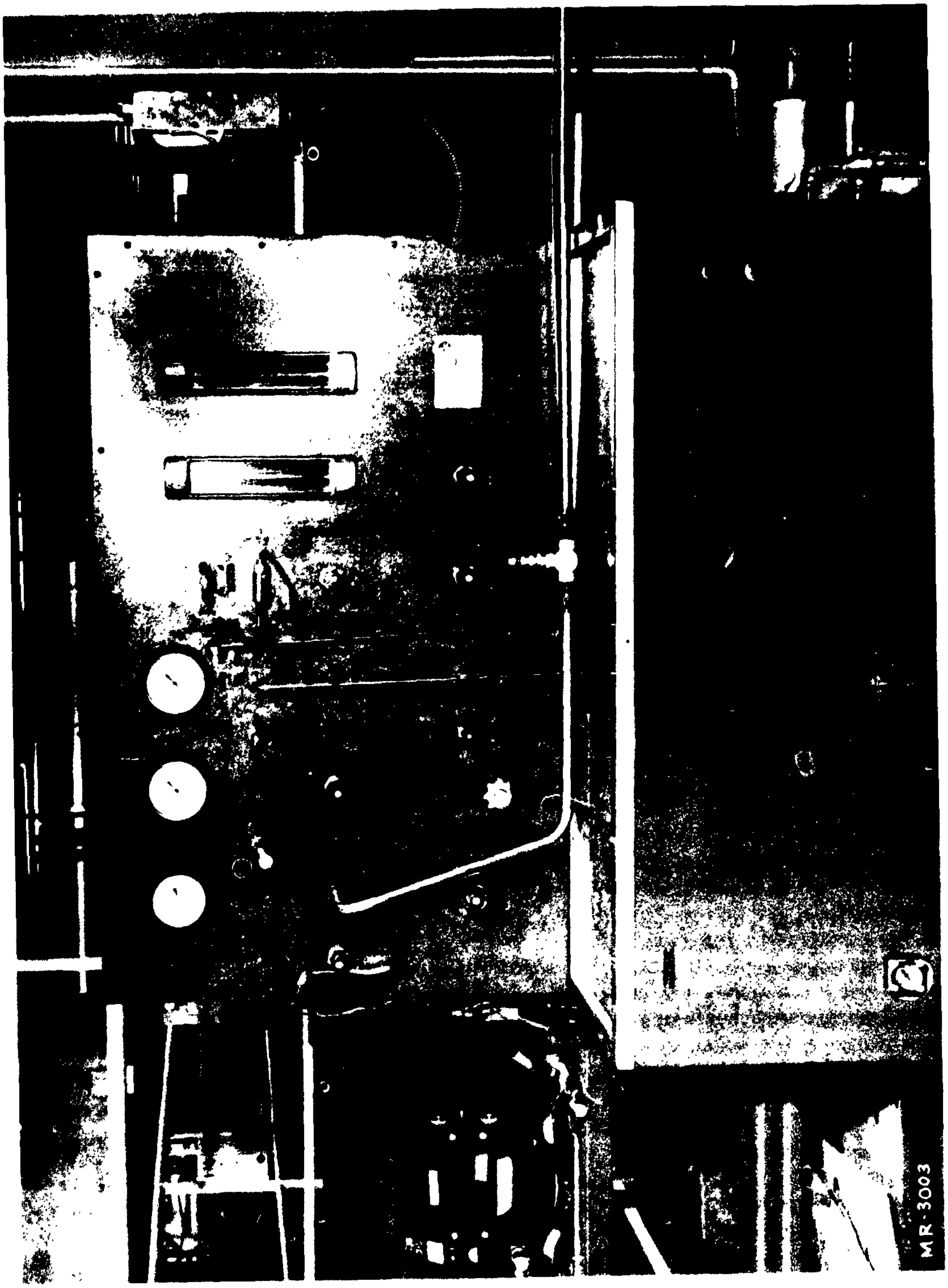


Figure 14. Hydraulic Test Console

Figure 15. Test Chamber and Test Console

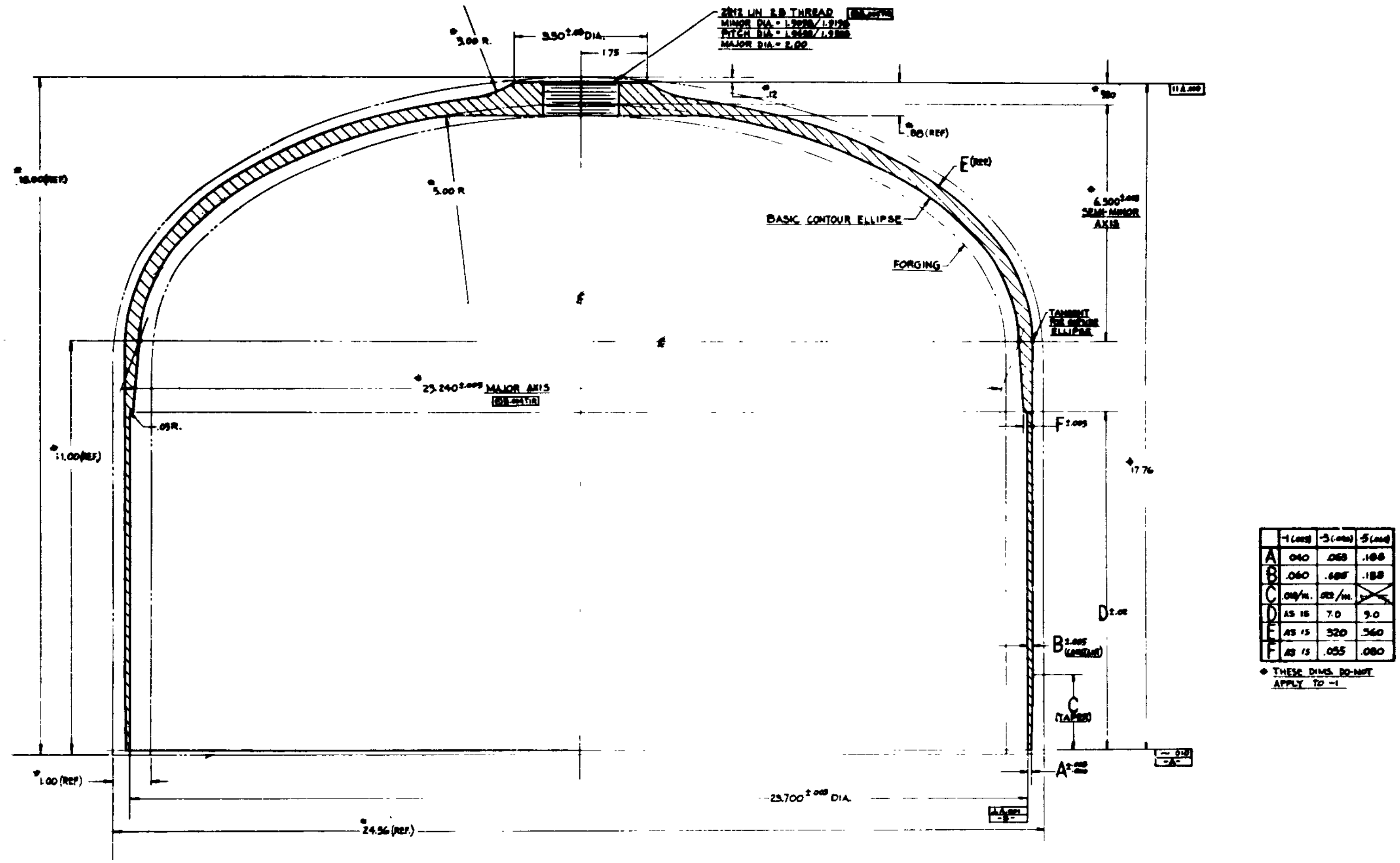


Figure 16. Header Configuration 244-1005

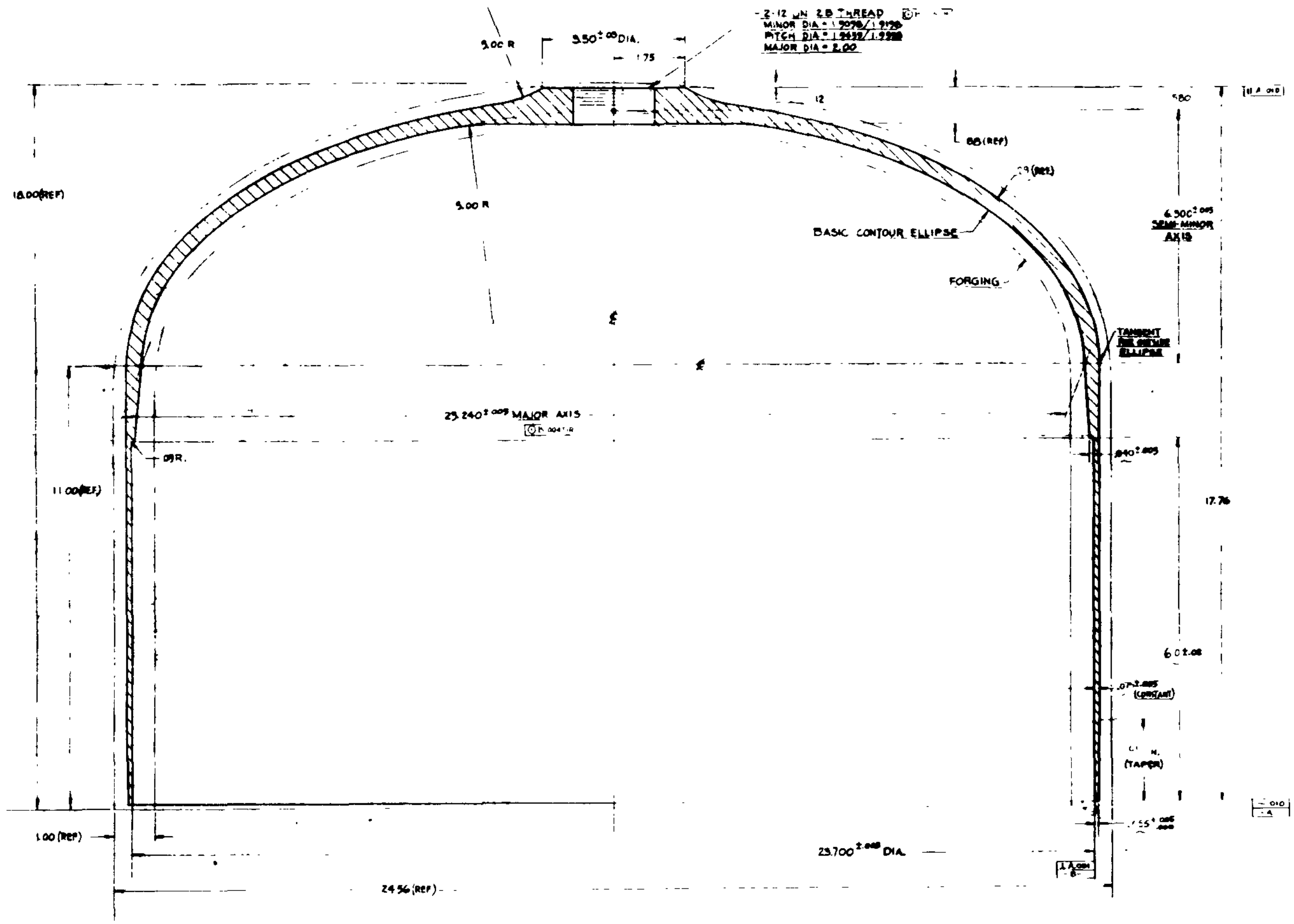
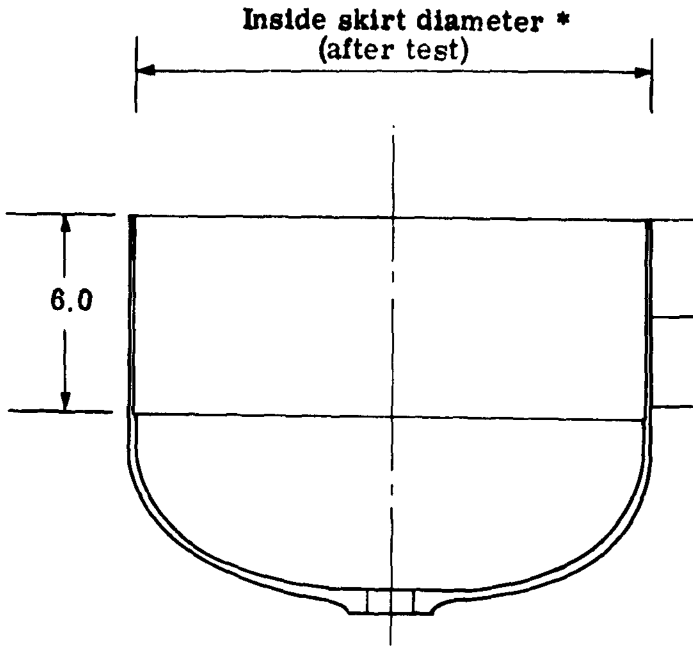
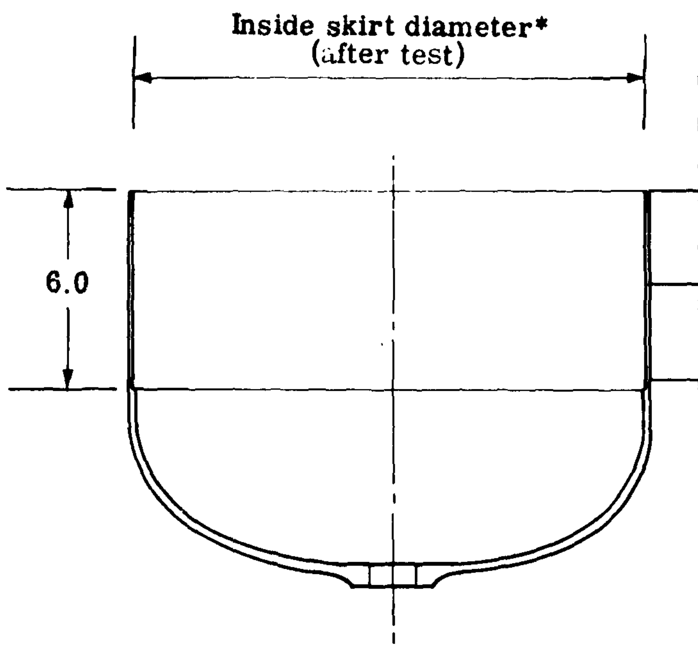


Figure 17. Header Configuration 244-1006



Diameter (inches)	
Station	Size
0.2	23.822
3.0	23.720
5.8	23.708

Header-244-1005-1



Diameter (inches)	
Station	Size
0.2	23.729
3.0	23.723
5.8	23.7[illegible]9

Header-244-1006-1

*Inside skirt diameter before test was 23.700 inches, constant.

Figure 18. Inside Skirt Diameter of Headers after Use in Test Number 1 (III-101)

design value because of decarburization sustained during previous use. The resulting edge thickness design was insufficient to withstand testing without excessive deformation at the free edge. The permanent set of the 244-1006-1 header is of the order of magnitude that was expected for the first pressure vessel test. In the manufacture of assembly III-102, extra thicknesses of adhesive were incorporated in the header joints. The fit-up interference between inner and outer rings was the minimum planned (0.010-inch diametric interference versus 0.020-inch nominal interference). When the vessel was tested, a leak developed between an inner and an outer ring at an internal pressure of 250 psi; the header joints remained intact. Examination of the FM47 bond line disclosed the adhesive had insufficient pressure during the cure cycle. After test, vessel 2 was disassembled for investigation; Figure 19 shows the detail parts. The dark area is the zone where no bond existed between inner and outer ring to allow the sealant to flow between the rings. Figure 20 is a close-up of the inner and outer rings that shows the unbonded area. Based on the examination after the test, it was judged that the FM1000 would have yielded a superior joint under similar bonding conditions because of its greater flow capability. The FM1000 adhesive was chosen for use with vessel 3, which was manufactured with the same gage material as vessel 2.

C. VESSEL 3

Pressure-vessel assembly 3 (V-103) was fabricated using the two headers of the 244-1005-3 configuration (see Figure 16), FM1000 adhesive, and five 0.021-inch-thick rings. The substitution of these headers, which were machined for use with the 0.038-inch-thick maraging steel rings, for the originally planned lighter headers (244-1005-1 and 244-1006-1) was done on the basis of the successful burst test of vessel 1 (III-101). Because the heavier headers (244-1005-3) were used, vessel 3 (V-103) had substantially the same discontinuity effect as that of vessel 1 (III-101); for vessel 1, a 0.064-inch-thick, 1.00-inch wide, 17-7PH reinforcement strap was used adjacent to one header with completely successful results (see "Fourth Quarterly Report," RAC 1160, Section III, page 13).

Because of the difficulties experienced with the assembly tooling (see Section IV) during the final assembly operation (ring-to-header joint), the edge of the ring was damaged. The damage consisted of a small permanent buckle approximately 1.0 inch wide by 0.25 inch long. This damage was discovered after the vessel had

Figure 19. Vessel III-102 Disassembled after Hydrostatic Test

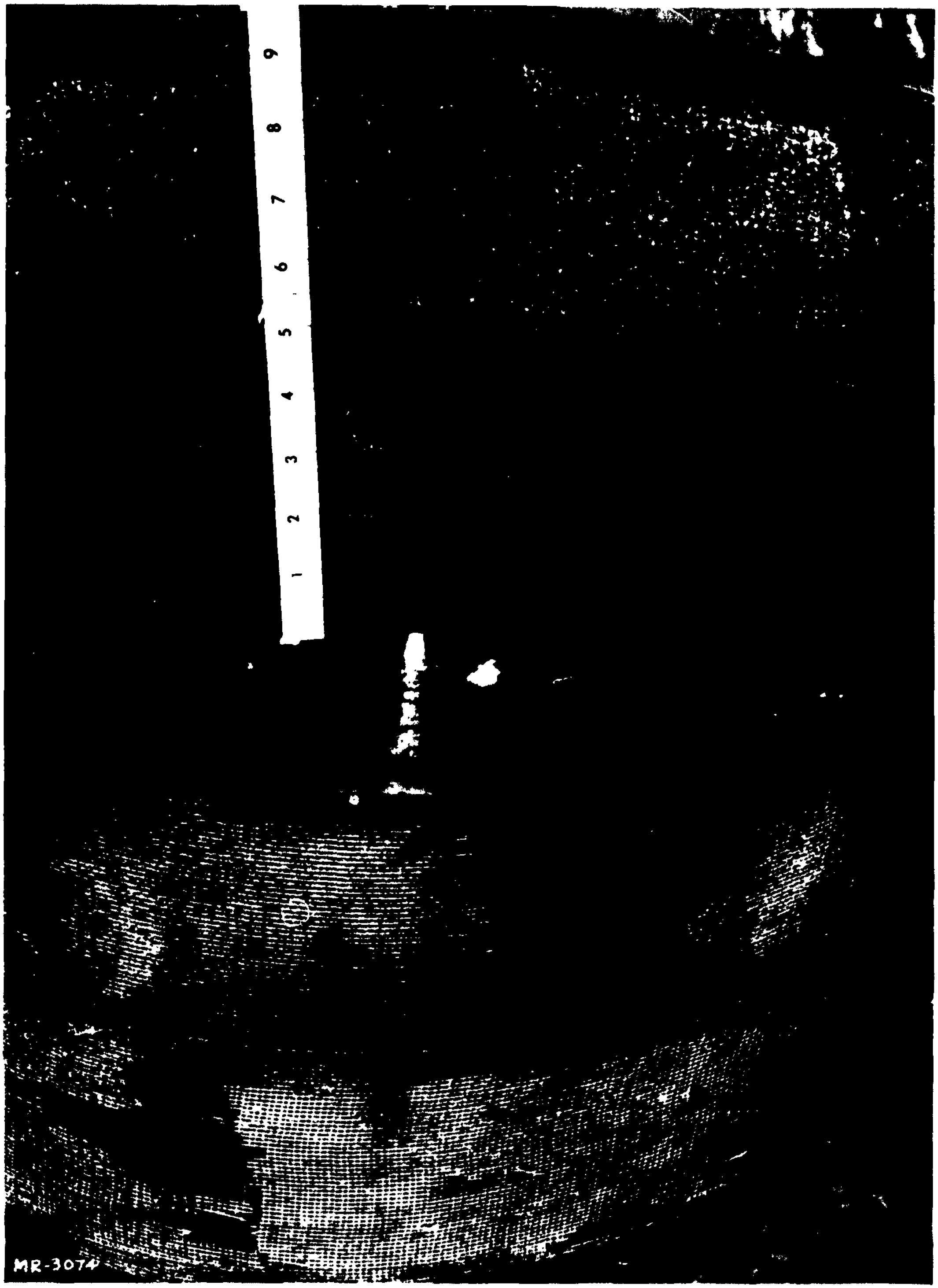


Figure 20. Close-up View of Failure Origin

been cured. The vessel configuration with closed domes at both ends (only 2.5-inch-diameter openings) makes inspection of the last joint very difficult. The vessel was disassembled by using hydraulic pressure to delaminate the damaged ring-to-header joint. The damaged edge of the ring was then trimmed to remove the buckled area. The header was then reassembled to the vessel, and the assembly was tested. The stress level reached during the delamination did not produce any discernible permanent effects on the vessel; in particular, the ring next to the header showed no effects. This was expected because the stress level reached was far below the yield strength of the metal parts.

The test resulted in failure of the parent sheet-metal material at 500 psi. This corresponds to a stress level of 140,000 psi in the material. The point of origin of this parent-metal failure was located and subjected to metallurgical examination. In general, the material appeared uniform in structure except for certain surface defects; the material had a hardness of approximately R_c 54. The defects observed were:

1) Grooves of 0.001- to 0.0015-inch depth on both surfaces; these grooves were caused by the mill processing

2) Localized areas on the surface of lower hardness that were 0.0002 inch thick

3) A single zone of light-etching, harder material (R_c 57 to 60)

The single zone also contained a 45-degree crack, which was 0.007 inch below the plane of the origin, that had propagated in this harder area and had extended 0.006 inch into the material. Polishing the specimen below the level of the crack resulted in the gradual disappearance of the harder zone and the crack; the grooves and softer surface areas persisted. It appears that the presence of the thin surface layer of softer material was not related to this failure. The failure was, therefore, attributed to the zone of harder, less ductile material in combination with the grooved condition of the surface. This defect was probably aggravated during the first pressure cycle used to delaminate the joint. Figure 21 shows the vessel just after the test. The failure is a longitudinal break completely around the periphery of the internal ring joining the header to the rest of the vessel. The metallurgical evaluation and the photomicrographs are presented in subsection II B of this report.

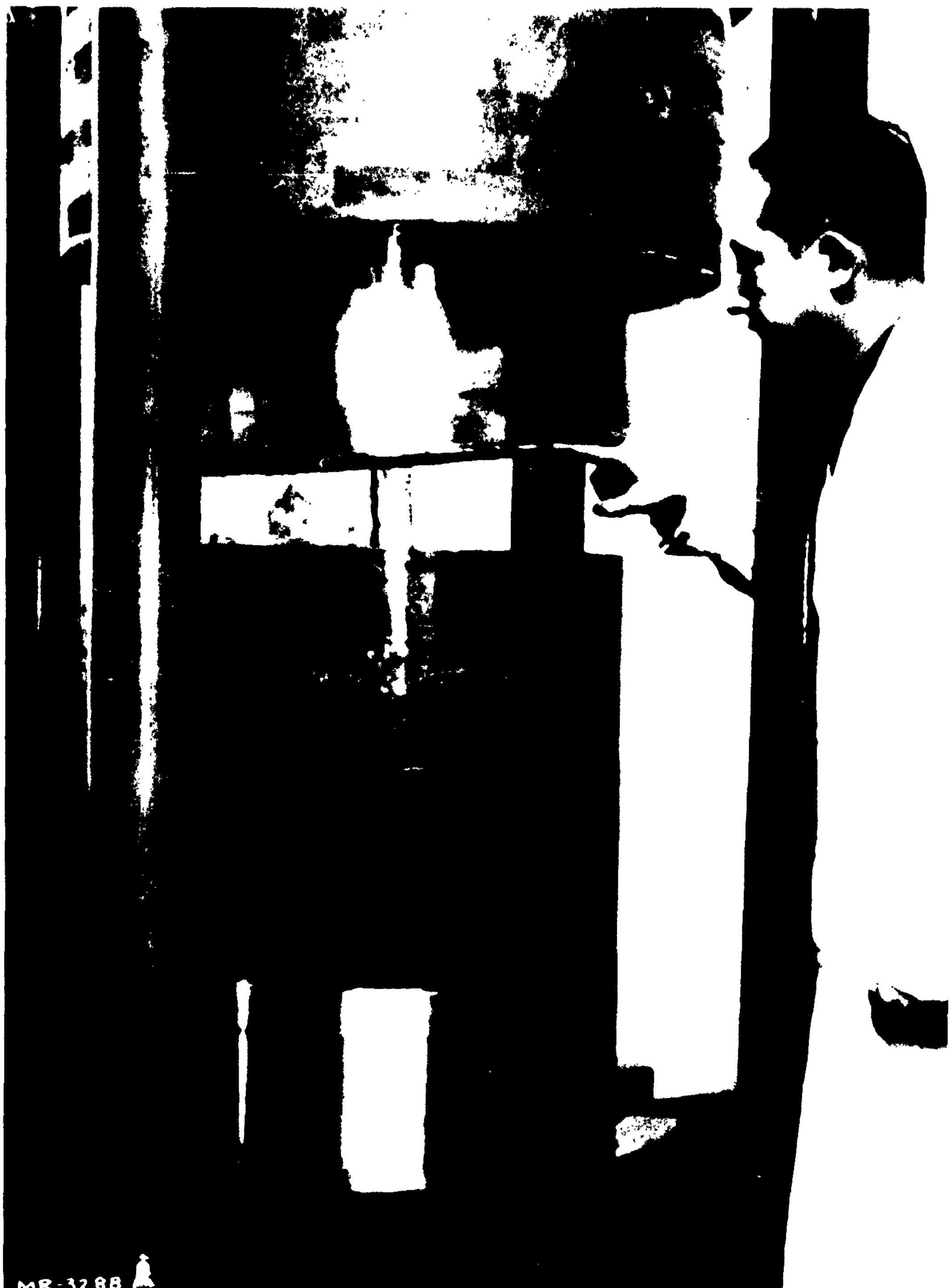


Figure 21. Vessel V-103 after Test

D. VESSEL 4

Assembly 4 (V-201) was fabricated using the two headers of the 244-1005-3 (see Figure 16) configuration, FM1000 adhesive, and five 0.038-inch-thick rings. The headers were not permanently deformed from their use in testing assembly V-103. When the vessel was hydrostatically tested, failure occurred at an internal pressure of 2030 psi. Figure 13 shows the pressure trace as it was recorded during this test. The ultimate membrane hoop stress in the metal rings was 320,000 psi based on the average thickness of the mar-aging rings (0.0375-inch average ring thickness). This stress level is approximately 12 percent greater than the uniaxial strength of the parent metal (see Table 6). Figures 22 and 23 show the metal parts just after removal from the test chamber and the original relative position of these parts. Examination of the parts showed that the failure originated in the parent metal and was not associated with any weldment or adhesive joint. A close-up view of the rings containing the failure origin is shown in Figure 24.

E. VESSEL 5

Pressure-vessel assembly 5 (V-202) was manufactured to the same configuration as assembly V-201 with one exception, which was necessitated by the permanent set sustained by the headers. Dimensional changes were made in the skirt portion of the headers because of the very high burst level of vessel V-201; these changes are shown in Figure 25. It can be seen that the magnitude of these changes is less than 0.1 percent of the permanent deformation except at the very edge where the tapered skirt is thinner to reduce the discontinuity effects in the mar-aging steel rings because of the increased thickness of the work-horse-design reusable headers. Because of the permanent set, extra layers of adhesive were used between the header skirt and the mar-aging steel inner-ring faying surfaces during the assembly process. When the assembly was tested, it failed at an internal pressure of 1520 psi. The failure originated in the weldment of the central inner ring at a membrane hoop stress level of 240,000 psi in the metal ring sections. To date, no uniaxial tensile tests have been performed on weldments in this gage of material, but data from tests on the 0.021- and 0.062-inch-thick welded specimens range from 243,000 psi to 265,000 psi (see "Fourth Quarterly Progress Report," Table 2, page 7). The average uniaxial strength of the parent material is 284,600 psi (see Table 6). Visual, X-ray, and stereomicroscopic examination of the weld

Figure 22. Vessel V-201 Parts Just after Removal from Test Chamber

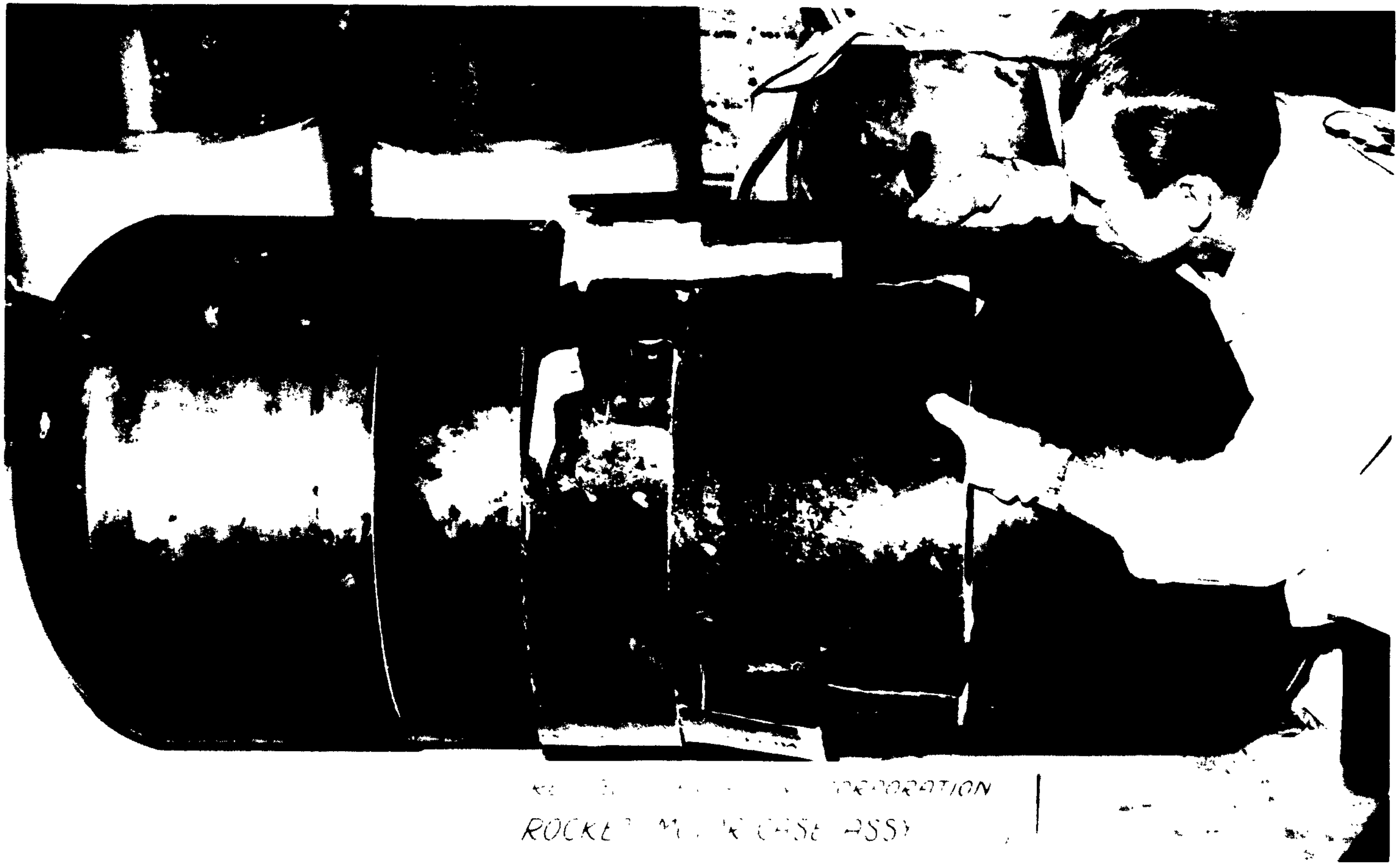


Figure 23. Parts Reassembled to Show Original Relative Position

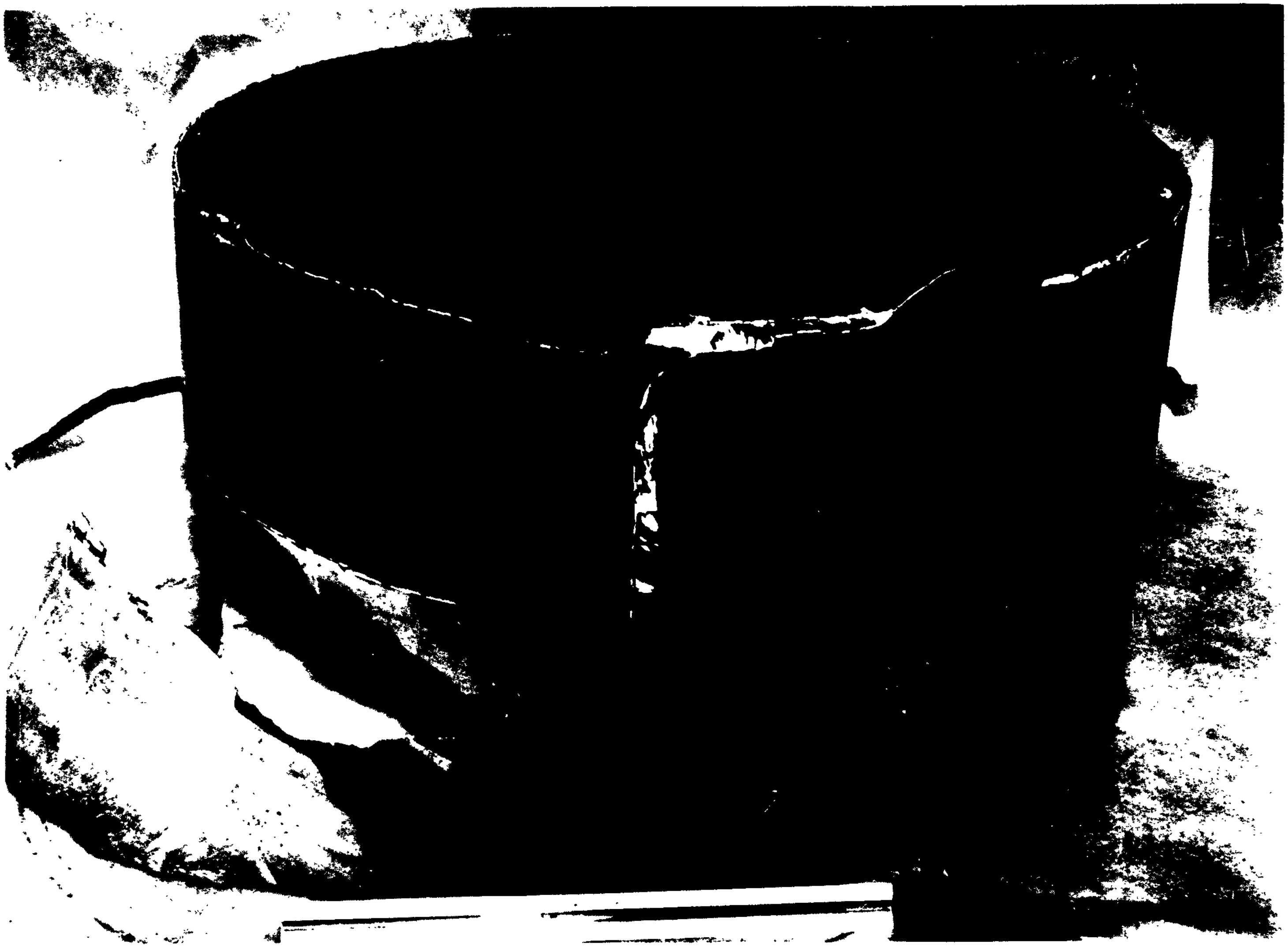
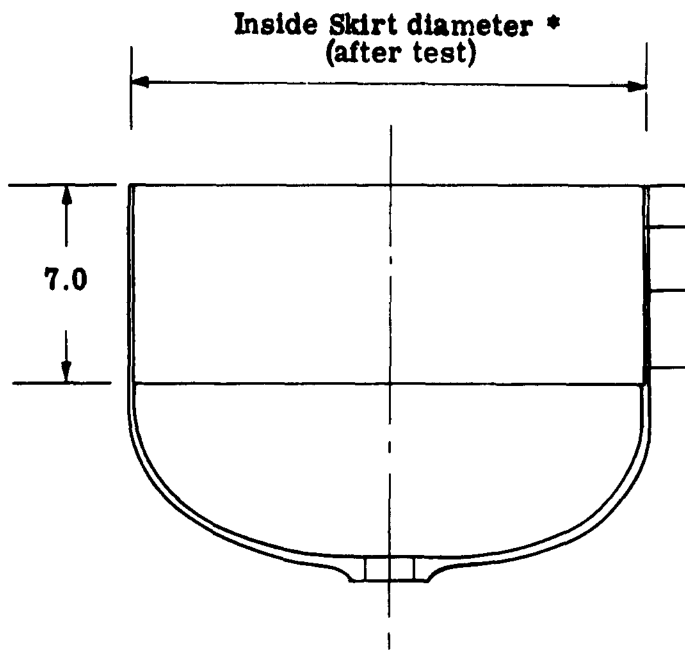
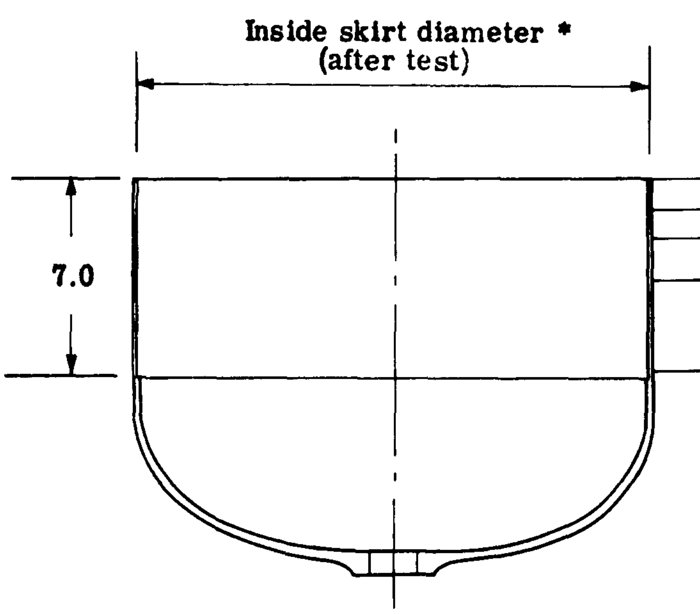


Figure 24. Close-up View of Failure Origin (V-201)



Diameter (inches)	
Station	Size
0.2	23.728
1.5	23.705
3.5	23.702
6.5	23.705

Header-244-1005-3A



Diameter (inches)	
Station	Size
0.2	23.727
1.0	23.717
2.0	23.712
3.5	23.707
6.5	23.705

Header-244-1005-3B

*Inside skirt diameter before test was 23.700 inches, constant.

Figure 25. Inside Skirt Diameter of Headers after Use in Test Number 4 (V-201)

revealed no unusual conditions. X-ray photographs of the weldment before and after failure are shown in Figures 26 and 27. Figure 28 shows an over-all view of the assembly after test; a close-up of the primary failure zone is shown in Figure 29. It is noteworthy that his view shows that the edge of the inner ring containing the weld failure remained bonded to the outside ring. Another view (Figure 30) of the reconstructed assembly shows the position of the weldments in the outer rings on the surface opposite the primary failure. The inner ring containing the weld along which the failure originated was delaminated; this ring is shown in Figure 31.

F. VESSEL 6

The last vessel tested during this quarter was pressure-vessel-assembly V-301. It was fabricated using the 244-1005-5 (see Figure 16) header configuration, FM1000 adhesive, and five 0.062-inch-thick mar-aging steel rings. Figure 32 shows the assembly being moved into the test chamber before the test. The vessel was hydrostatically tested to failure at an internal pressure of 2290 psi. The failure was caused by the delamination of one of the adhesive-bonded, header-to-mar-aging-steel-ring joints. Examination of the faying surfaces subsequent to failure disclosed an unbonded zone equaling approximately 20 percent of the total joint area. Figure 33 shows the vessel after failure; the dark area at the top of the joint is the unbonded zone. Close-up views of the failure origin on the header and ring faying surfaces are shown in Figures 34 and 35. The longitudinal cracks seen in the steel ring were caused by the pressure exerted on the single thickness of inner ring as the header moved off the cylinder on a skewed angle. Figures 36 and 37 show the areas on the header and steel inner ring that sustained the end load until failure occurred. The strain striations on the adhesive are typical of adhesive failures where the adhesive has performed its function of load transfer.

Preliminary evaluation of this failure indicates that the lack of bond over this large area was caused by the stiffness of the work-horse header skirt. This situation was the result of attempting to use two ideas in one design. The strength requirement for the D6-A steel headers (244-1005-5) made it necessary that the skirt section (0.188 inch thick) be nine times stiffer than the mar-aging steel ring to which it is bonded. The flexural and natural arch rigidity of the thick-walled, cylindrical skirt of the header prevented the proper mating with the lighter, more flexible, 0.062-inch-thick rings during the adhesive cure cycle. For optimum

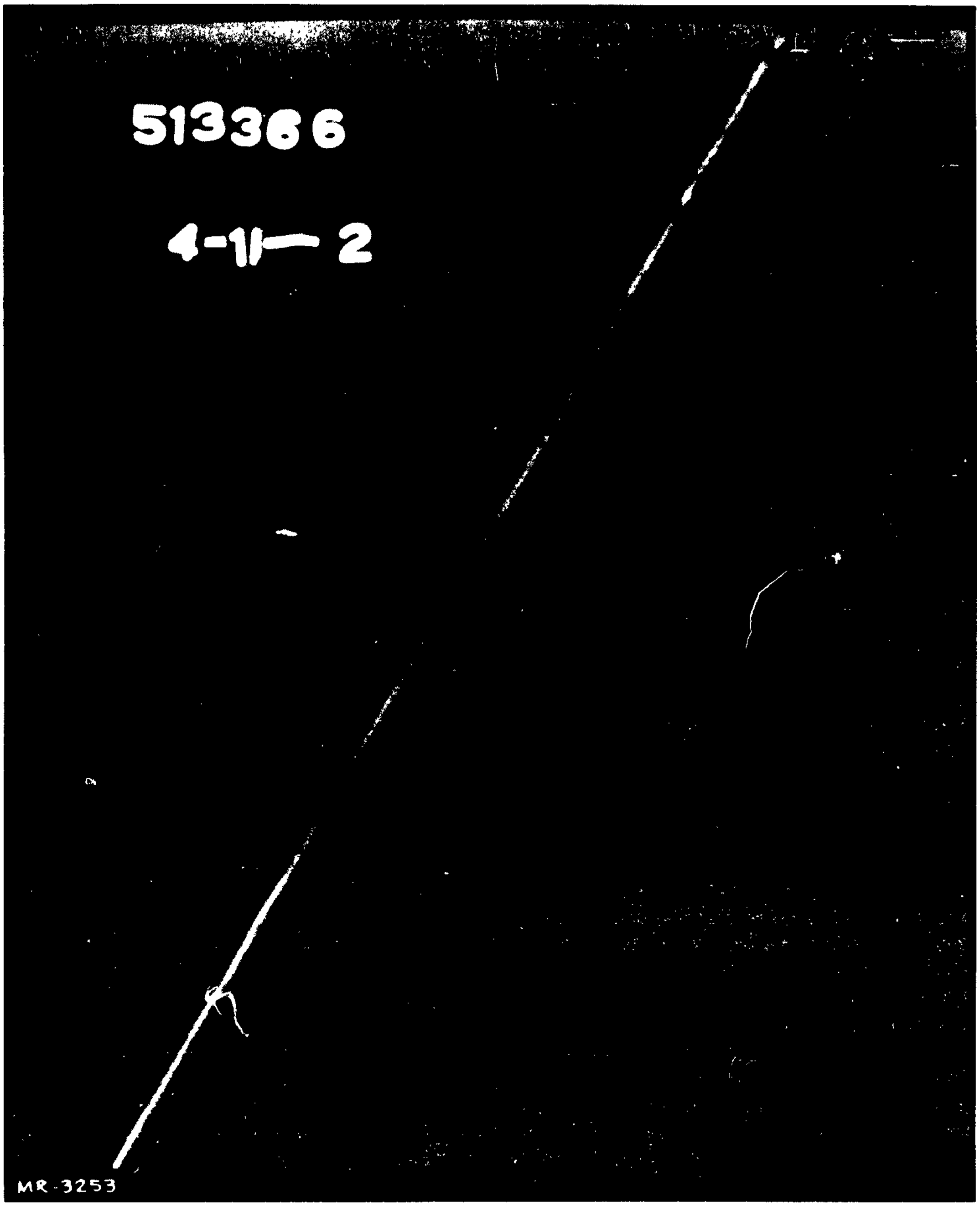


Figure 26. X-ray of Weldment in Ring before Test (V-202)

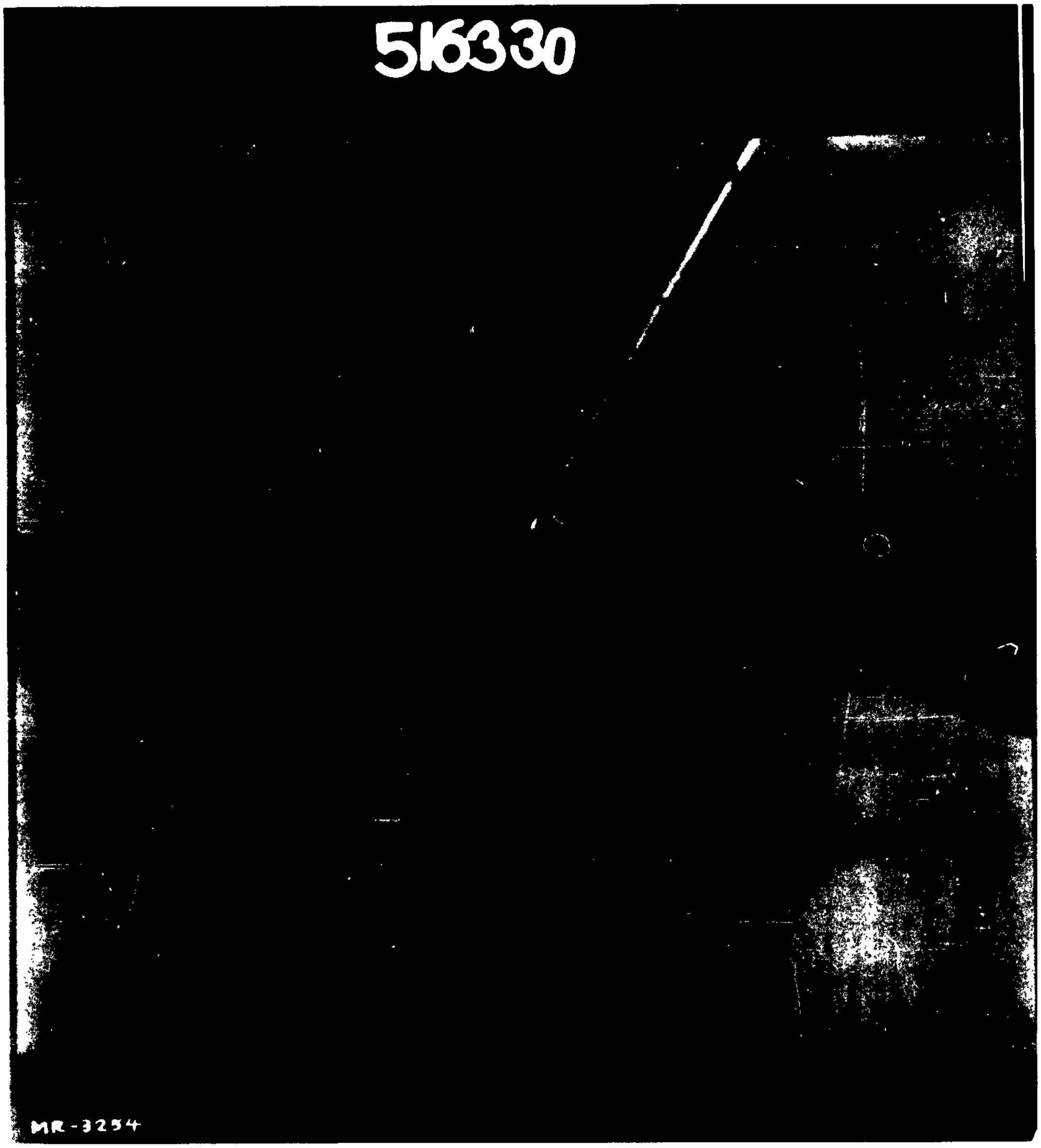


Figure 27. X-ray of Weldment after Hydrostatic Test of Assembly V-202

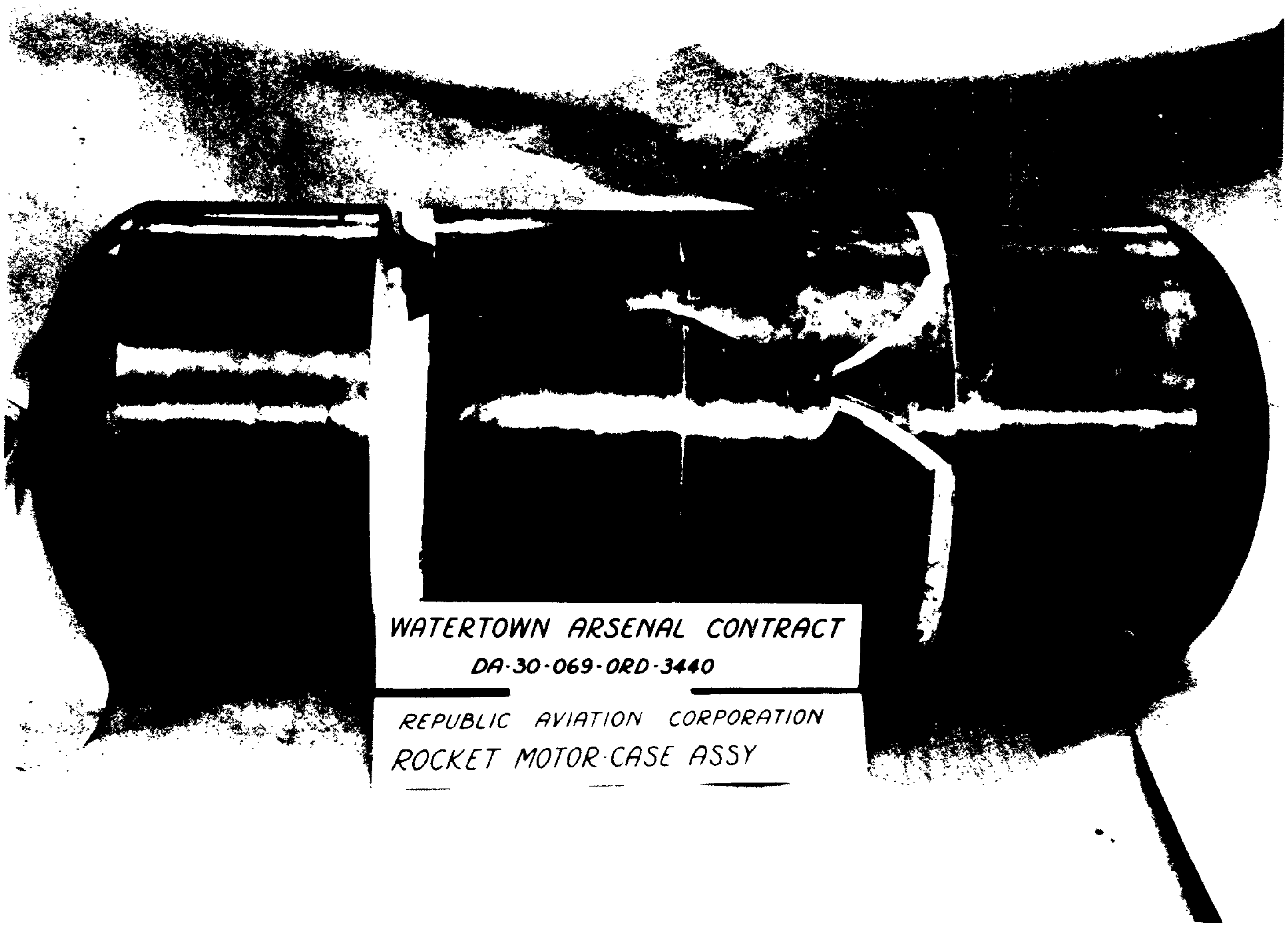


Figure 28. Reassembled Parts of Vessel V-202 after Hydrostatic Test

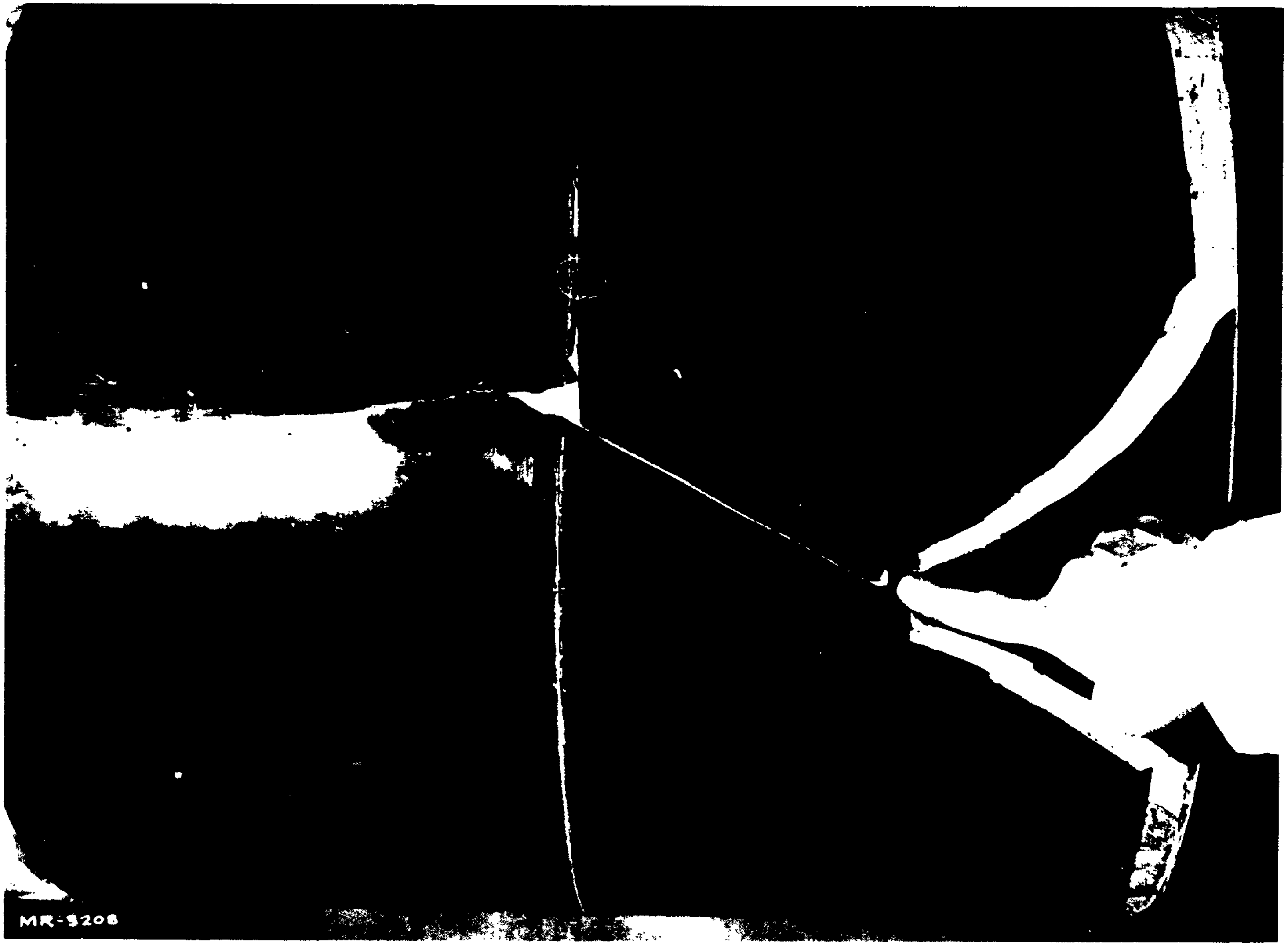


Figure 29. Close-up of Primary Failure Zone

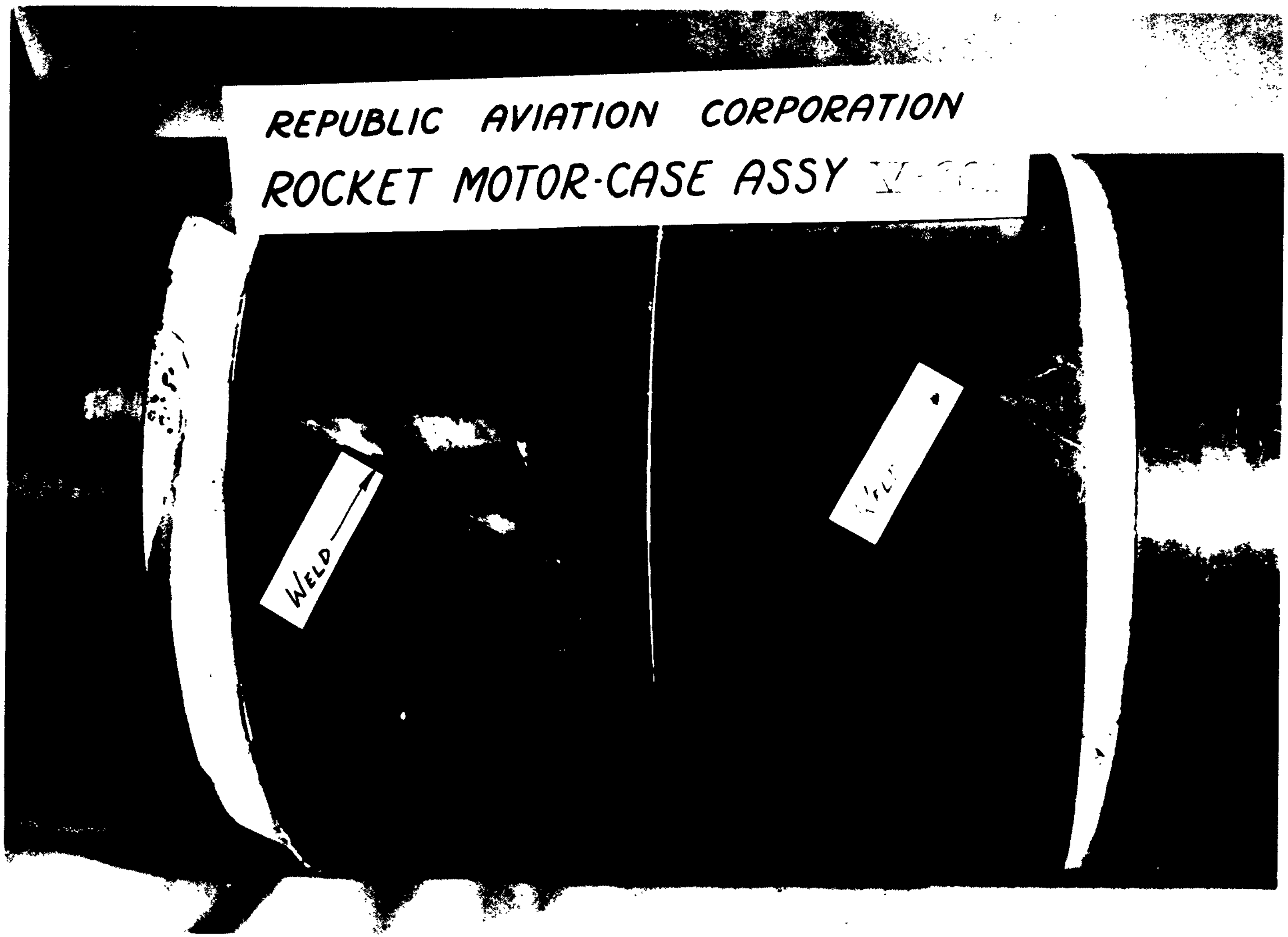


Figure 30. View of Reassembled Parts

Figure 31. Central Inner Ring

Figure 32. Vessel V-301 Being Placed in Test Chamber

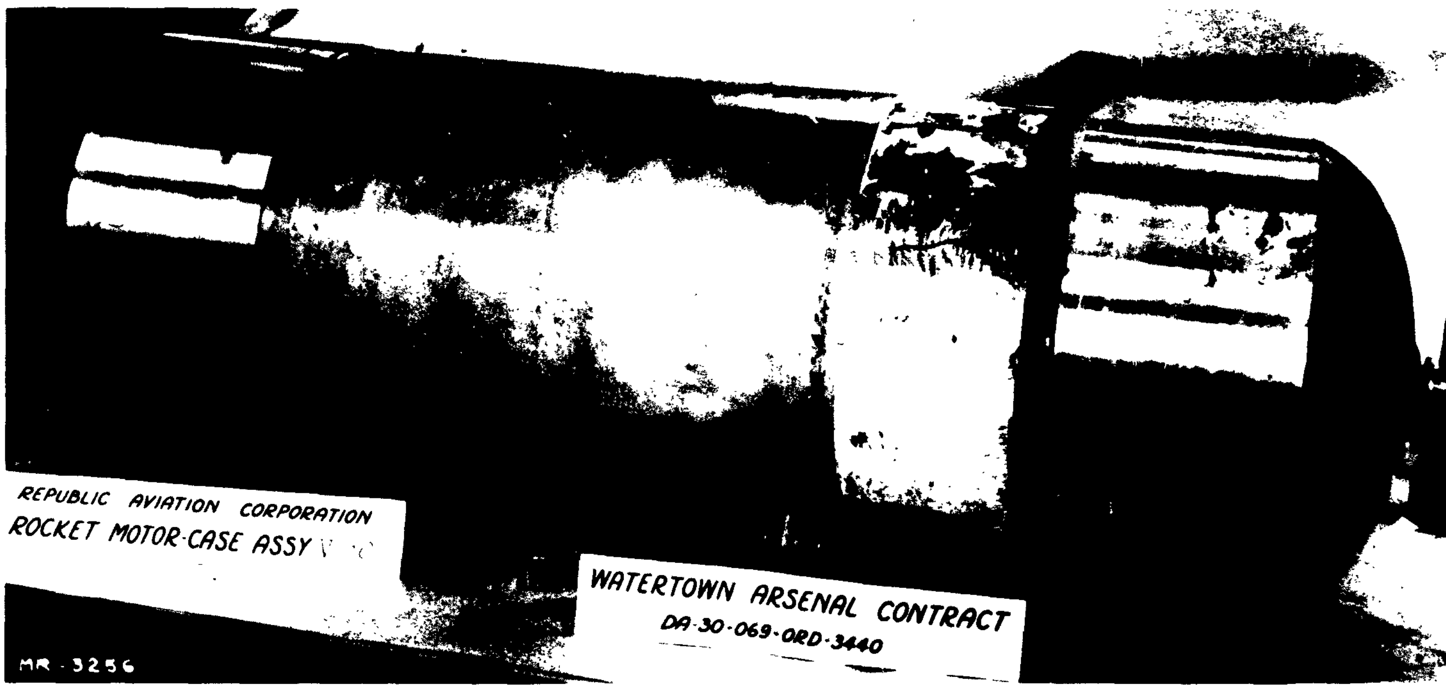


Figure 33. Vessel V-301 after Hydrostatic Test

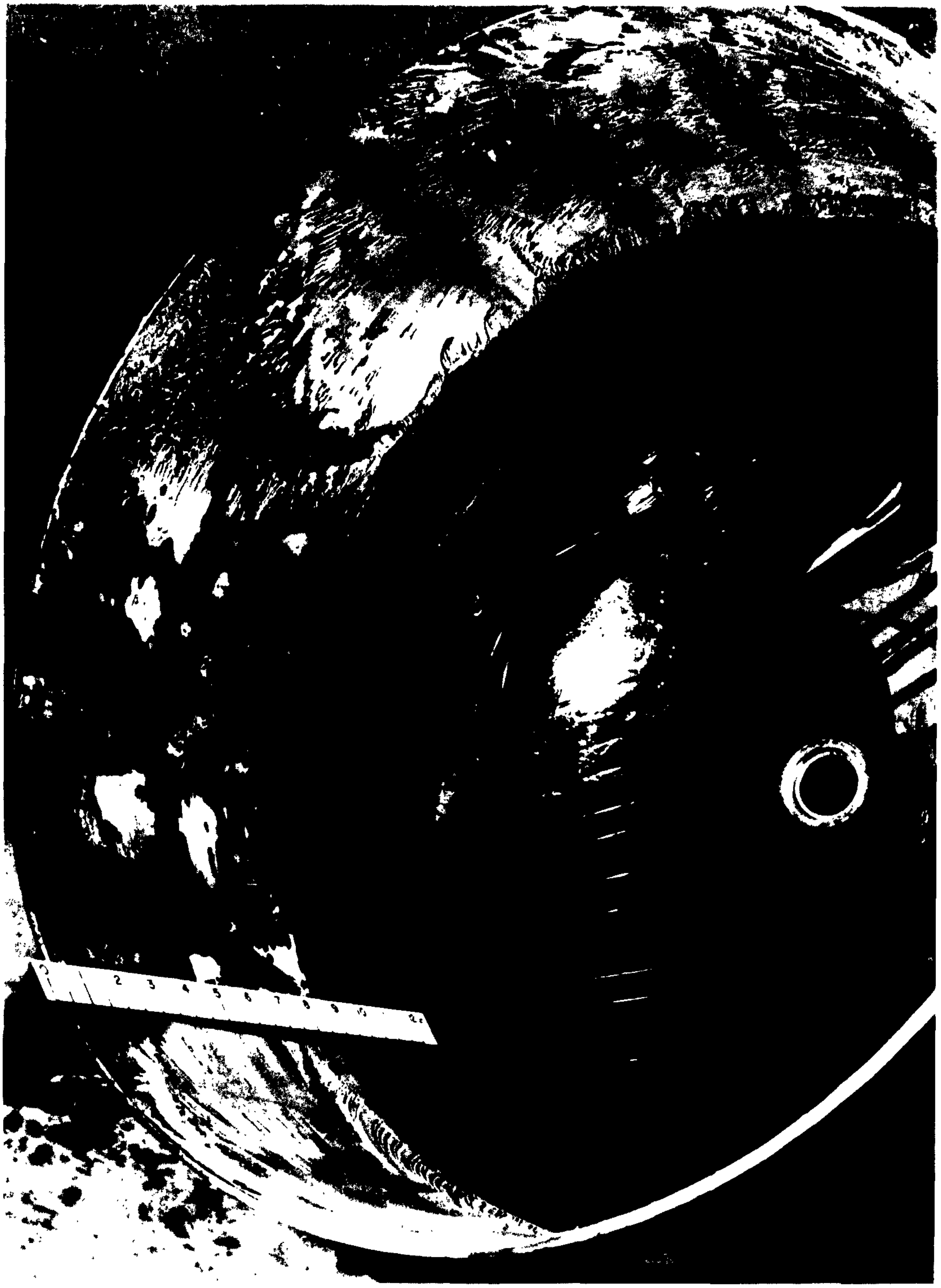


Figure 34. Close-up of Failure Origin on Header (V-301)

Figure 35. Close-up of Failure Origin on Inner Ring (V-301)

Figure 36. Close-up of Header Adhesive Joint Opposite Failure (V-301)

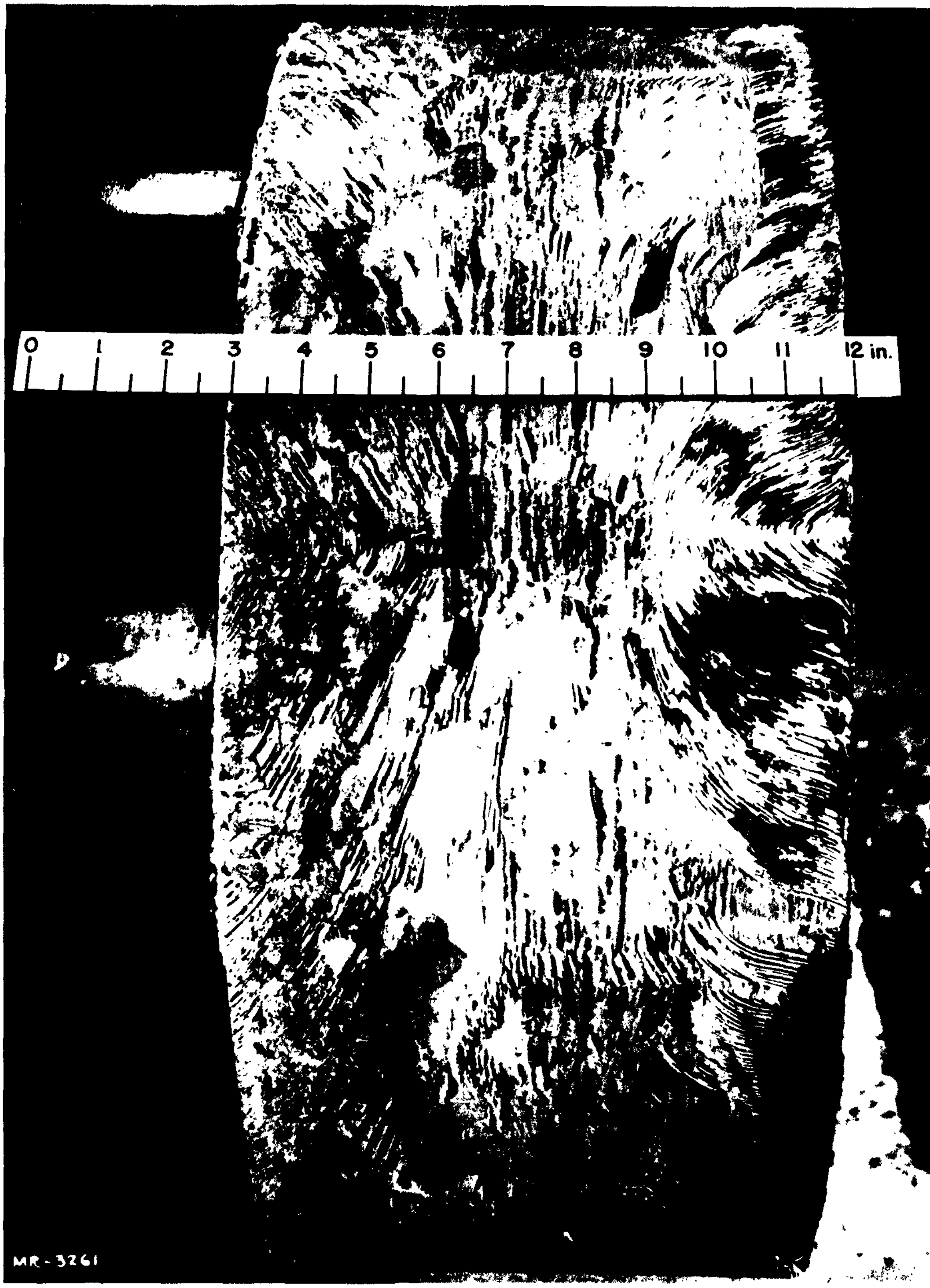


Figure 37. Close-up of Inner Ring Adhesive Joint
Opposite Failure (V-301)

design, a header fabricated especially for use with each assembly would have the same thickness and rigidity as the ring section to which it is bonded. If this were done, the poor contact pressure in the local area during cure would not occur under the fit-up conditions that are imposed (line-to-line diametric fit with the tolerance on the side of interference) during assembly.

SECTION IV

TOOLING AND MANUFACTURING RESULTS

The assembly process, as originally planned, consists of heating the outer rings and headers by means of heating blankets in order to increase their diameter to overcome the interference fit of the metal parts and adhesive. This permits their assembly over the inner rings on which the adhesive has been positioned. The resulting assembly is then cured in an oven in accordance with the requirements of the adhesive used.

During the assembly of pressure vessels III-101, III-102, and V-103, the operational characteristics of the bonding assembly fixture proved to be unreliable and, in fact, caused a header to bind 1.00 inch before reaching its proper position in assembly III-101 (see "Fourth Quarterly Progress Report," Section IV). It also caused the small local permanent deformation that necessitated the disassembly of vessel V-103 (subsection III B). The difficulties with this approach were:

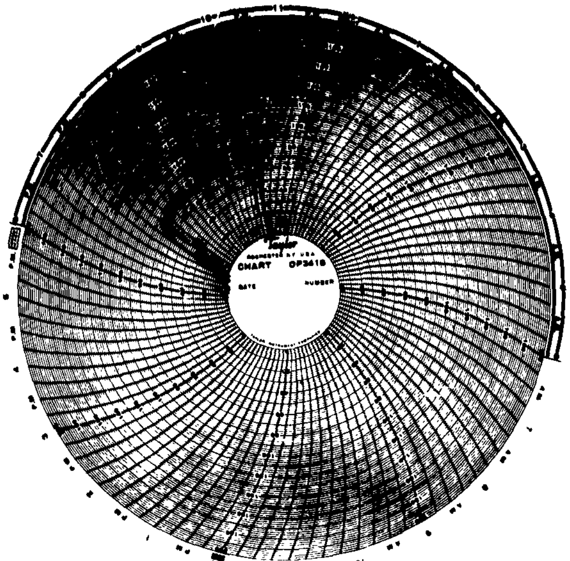
1) The small (heat slip) diametric clearance, due to the allowable thermal input and the diameter of the vessel, made it necessary to have very accurate alignment and retention of the circular contour of the sheet-metal parts. This procedure becomes more feasible as diameter increases (clearance due to temperature differential increases directly with diameter) because the alignment requirements are less stringent

2) The radial drill that was used to provide the alignment was not able to maintain sufficient accuracy with respect to concentric alignment

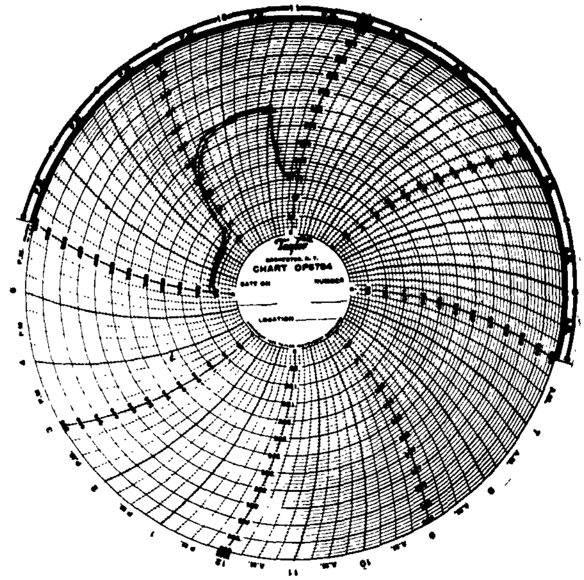
3) The loss of heat because of radiation was a problem with respect to maintaining sufficient temperature in the outer rings and headers to keep the clearance throughout the assembly while keeping the inner ring cool enough to prevent tacking of the adhesive before completion of the operation

4) The flexibility of the rings required more elaborate tooling to maintain the circular shape of the outer (hot) rings so that the clearance would not be reduced during the heat slip-assembly process

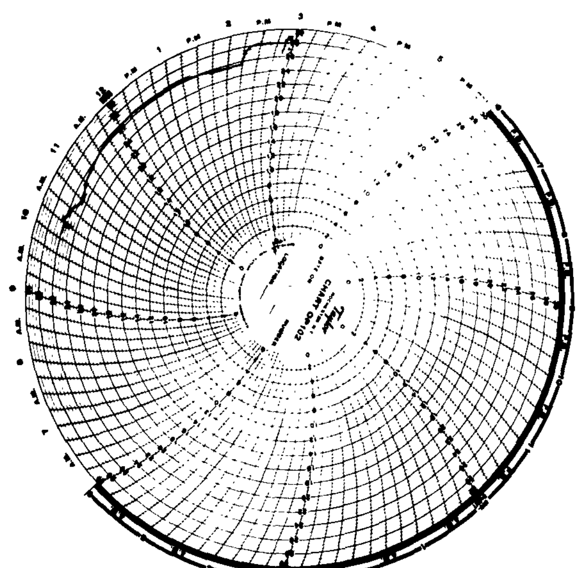
Because of the difficulties encountered and because of the extensive tooling modifications that would be required to succeed with the heat assembly process, an alternative method of manufacture was developed. The assembly method was modified to reduce the diametric interference of the metal parts and adhesive to range from line-to-line fit to a maximum of 0.008-inch diametric interference. The adhesive is fixed to the inner rings by vacuum bag molding at 150 degrees F for 1 hour. The outer rings and headers are forced over the inner rings by a mechanically applied driving force at room temperature. The slight diametric interference is overcome primarily by elastic deformation in the uncured adhesive. The assembly is then vacuum bagged and autoclave molded at 325 to 350 degrees F for 1 hour under pressure appropriate for the thickness of the mar-aging sheet-metal rings (0.021-inch rings, 45 psi; 0.038-inch rings, 80 psi; 0.062-inch rings, 110 psi). Figure 38 shows the pressure and temperature cycle sustained by vessel V-202 during the autoclave molding cycle. As a last step, the vessel is then coated internally with a neoprene rubber compound as a sealant and baked at 150 degrees F for 3 hours.



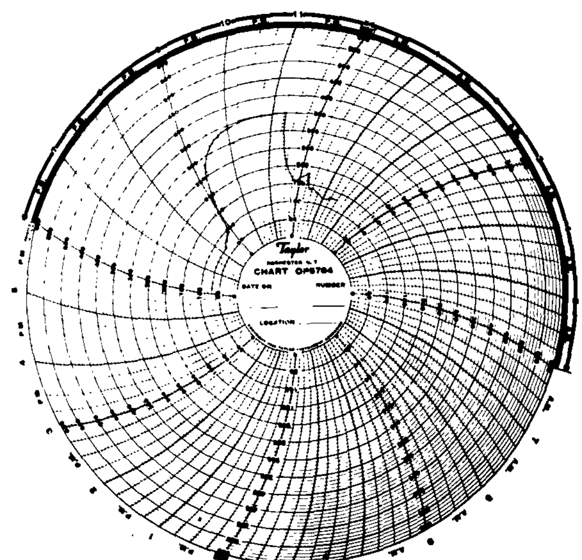
a. Pressure (psi)



b. Temperature (degrees F), Thermocouples on Vessel



c. Vacuum (inches of mercury



d. Autoclave Temperature (degrees F)

Figure 38. Autoclave Temperature and Pressure Charts (V-202)

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Office of Naval Research
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